

August 21, 2017

Ms. Sonia Vega On-Scene Coordinator U.S. Environmental Protection Agency Region 5 600 A Joliet Road Willowbrook, Illinois 60527

Subject: Final Sampling and Analysis Plan

Niagara LaSalle – Optima Steel RS Hammond, Lake County, Indiana

EPA Contract No. EP-S5-13-01 (START IV, Region 5)

EPA TDD No. 0001-1707-004

Document Tracking No. (DTN): 1993

Dear Ms. Vega:

The Tetra Tech, Inc. Superfund Technical Assessment and Response Team (START) is submitting the Final Sampling and Analysis Plan (SAP) for the Niagara LaSalle – Optima Steel Site in Hammond, Lake County, Indiana. The Final SAP summarizes soil sampling activities to take place at an active industrial property and surrounding residential area to assess lead contamination related to steel processing.

Please call me at (312) 201-7721 if you have any questions or comments regarding this submittal.

Sincerely,

Rachel Houle Project Manager

Enclosures (1)

cc: TDD file

Kevin Scott, Tetra Tech

FINAL SAMPLING AND ANALYSIS PLAN

NIAGARA LASALLE – OPTIMA STEEL SITE HAMMOND, LAKE COUNTY, INDIANA

Prepared for

U.S. ENVIRONMENTAL PROTECTION AGENCY Region 5 Chicago, Illinois 60604



TDD#:	S05-0001-1707-004	
EPA OSC:	Sonia Vega	
SITE NAME:	Niagara LaSalle-Optima Steel RS	
SITE LOCATION:	Multiple Locations, Hammond, Indiana	
SAMPLING ACTIVITIES:	Surface and Subsurface Soil Sampling	
SAMPLING DATES:	Multiple Dates, August and September 2017	
SAP PREPARER:	Rachel Houle	
SIGNATURE/DATE	Rfford	08/21/2017
QC REVIEWER:	John Dirgo	
SIGNATURE/DATE:	John Reign	08/21/2017
EPA OSC APPROVAL		
SIGNATURE/DATE:		
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1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) identifies the data collection activities and associated quality assurance/quality control (QA/QC) measures specific to the Niagara LaSalle-Optima Steel Site located in Hammond, Lake County, Indiana (Appendix A, Figures 1 and 2). The site-specific sampling, analytical, and QA/QC procedures described in this SAP are designed to accommodate the project scope of work and requirements requested by the U.S. Environmental Protection Agency (EPA).

2.0 SCOPE OF WORK

Under Technical Direction Document (TDD) S05-0001-1707-004, EPA Region 5 tasked the Tetra Tech Superfund Technical Assessment and Response Team (START) to assist with sampling at the site. The overall goal of the sampling effort is to assess levels of contamination in soils on the Niagara LaSalle-Optima Steel property caused by steel processing at the site.

The EPA On-Scene Coordinator (OSC) tasked START with the following objectives:

- Accompany EPA on a site reconnaissance to identify potential sources of contamination and to select sampling locations and depths.
- Collect up to 10 surface and subsurface soil samples; field-screen the soil collected with an X-ray fluorescence (XRF) analyzer; and select samples for laboratory analysis. The exact number of samples will be determined after the site reconnaissance.
- Select a subset of soil samples for toxicity characteristic leaching procedure (TCLP) analysis of metals.
- Photo-document sampling activities and sampling locations.
- Collect subsequent residential soil samples, contingent on the initial soil sampling results.

This SAP describes the field screening, sampling, analytical, and QA/QC requirements for activities at the site. Figures and tables for this SAP are provided in Appendices A and B. The figures will be amended, in some copies of this SAP distributed, to protect the confidentiality of the property owners. START standard operating procedures (SOP) to be used during this investigation are provided in Appendix C.

3.0 PROJECT TEAM

The personnel listed in the table below will be involved in planning or technical activities for this site. The OSC and each member of the field team will receive a copy of the SAP, and a copy will be retained in the site file. Copies of this SAP distributed outside of EPA and its contractors will be amended to protect the confidentiality of the property owners.

Personnel	Title	Organization	Phone Number	Email
Sonia Vega	OSC	EPA	630.481.5025	Vega.Sonia@epa.gov
Rachel Houle	Project Manager	START	708.955.4569	Rachel.Houle@tetratech.com
John Dirgo	QA Manager	START	312.201.7765	John.Dirgo@tetratech.com
Rachel Houle	Field Staff	START	708.955.4569	Rachel.Houle@tetratech.com

Notes:

EPA = U.S. Environmental Protection Agency

OSC = On-Scene Coordinator

QA = Quality assurance

START = Superfund Technical Assessment and Response Team

4.0 SITE LOCATION AND DESCRIPTION

The site, which is located in Hammond, Lake County, Indiana, consists of the active Niagara LaSalle-Optima Steel property located at 1412 East 150th Street, and nearby residential properties bounded by 150th Street to the north, the Indiana Toll Road to the east, the Niagara LaSalle property to the south, and Magnolia Avenue to the west.

Niagara LaSalle is an active cold-finished steel bar producing plant. Aerial photographs of the plant show red staining on the northern part of the property near the baghouse dust collector and on the north adjacent street and sidewalks. XRF screening conducted by the Indiana Department of Environmental Management (IDEM) in April 2017 found concentrations of lead as high as 4,822 parts per million (ppm) on the plant property and as high as 3,468 ppm within the surrounding residential areas. IDEM noted that lead concentrations may actually be higher because of the high moisture content of the soil on the day of the XRF screening. Laboratory analysis of soil samples was recommended to obtain more accurate results.

5.0 PROPOSED SCHEDULE

Sampling activities are scheduled for August 2017. The preliminary laboratory results for total and TCLP metals are anticipated to be available 2 weeks to 1 month after sampling. At the latest, these results will be available in September 2017.

All laboratory analytical data will be validated by a START chemist when the full laboratory reports become available. The validated analytical results and other findings will be provided to EPA in a Site Assessment Report. The anticipated schedule is outlined in the table below.

PROPOSED SCHEDULE

	Dates (Mont	h, Day, Year)		
Activities	Anticipated Date of Initiation	Anticipated Date of Completion	Deliverables	Deliverable Due Date
HASP Preparation SAP Preparation	July 24, 2017	August 1, 2017	HASP, SAP	August 2, 2017
Sample Collection	August 3, 2017	August 3, 2017	Log Books, Sampling and Screening Logs	Not applicable
Laboratory Analysis	August 2017	September 2017	Laboratory Analytical Reports	4 weeks after submittal of samples
Data Validation	September 2017	September 2017	Data Validation Reports	1 week after receipt of the final laboratory analytical report
Subsequent Residential Sampling, Laboratory Analysis, and Data Validation	September 2017	TBD	Log Books, Sampling and Screening Logs; Laboratory Analytical Reports; Data Validation Reports	TBD
Draft Project Report	TBD	TBD	Draft Site Assessment Report	2 weeks after the data validation reports are received
Final Project Report	Upon receipt of comments to the draft report	One week after receipt of client comments	Final Site Assessment Report	TBD

Notes:

HASP = Health and Safety Plan SAP = Sampling and Analysis Plan TBD = To be determined

6.0 SAMPLE LOCATIONS AND COLLECTION PROCEDURES

Starting on August 2, 2017, EPA and START will conduct a site visit and assessment by completing a facility walk-through and collecting soil samples on the Niagara-LaSalle property. The sampling locations will be selected during the site reconnaissance and will be logged with photographs and in the logbook. Global positioning system (GPS) coordinates of sampling locations will be recorded using a Trimble GPS unit.

EPA anticipates the need for subsequent XRF screening and soil sampling at the residential properties surrounding the Niagara-LaSalle site. The properties to be sampled will be selected based on: (1) signed access agreements, and (2) distance of the property from the Niagara-LaSalle facility. The site assessment will be conducted by following the "Superfund Lead-Contaminated Residential Sites Handbook" as guidance (EPA 2003). The number of residential properties will be determined after canvassing the neighborhood to assess the voluntary interest of residents in having their soil sampled. Section 6.1 discusses residential sample locations based on property type. Section 6.2 describes the sample collection process.

6.1 RESIDENTIAL PROPERTIES

According to the "Superfund Lead-Contaminated Residential Sites Handbook," residential properties are considered to be single and multi-family dwellings, apartment complexes, vacant lots in residential areas, schools, day-care centers, playgrounds, parks, and greenways. The sample design for the residential areas varies depending on the position of the house and the size of the lot. The Niagara-LaSalle Site investigation area includes single-family residences. The following subsections discuss sample design for single-family residences.

6.1.1 Single-family Homes

Based on a review of aerial images of the investigation area and the Lake County Surveyor GIS website (Lake IN GIS Portal), it appears that single-family homes inside the investigation area are typically situated on lots that are approximately 5,000 square feet or less, with no significant side yard. According to the "Superfund Lead-Contaminated Residential Sites Handbook," three samples should be collected at single-family lots that are less than 5,000 square feet with no significant side yard: one sample each from the front yard of the property; one from the back yard of the property; and one from the drip zone surrounding the residential property. Additional samples may be collected based on property-specific features.

The front yard and back yard samples will consist of a five-point composite soil sample. Generally, sample aliquots will be collected from the center of each yard and from points that area approximately half-way between the center and each corner of the yard. The five aliquots for each sample will be collected from the same depth interval.

At least one drip zone sample will also be collected from each single-family property. The drip zone sample will consist of at least four aliquots. An aliquot will be collected from approximately the center of each side of the residence, at a distance of approximately 6 to 30 inches from the exterior walls.

Additional aliquots may be collected based on features observed in the field, such as bare spots, differences in house exterior, or areas where runoff accumulates. If gutter downspouts are observed to discharge directly to the ground surface, an additional aliquot may be collected from the area near the downspout discharge to account for fine material deposited by flow through the downspout. Aliquots will be collected from 0 to 6 inches below ground surface (bgs).

In addition to the standard composite sample locations discussed above, a distinct play area or garden at a single-family lot will also be sampled. A five-point composite sample methodology will be followed in these areas, as long as space and existing infrastructure allow.

6.2 SAMPLE COLLECTION PROCEDURES

The sampling locations and depths for the Niagara LaSalle property will be selected after the site reconnaissance on August 2, 2017. Each discrete surface and subsurface soil sample will be collected and screened off site for lead and arsenic content using an XRF analyzer. At EPA's discretion, and as determined by XRF results, a number of samples will be submitted to a Tetra Tech-procured laboratory to be analyzed for total metals, and a subset of these samples will be analyzed for TCLP metals. All sampling activities will be documented in a field logbook as well as photo-documented, in accordance with Tetra Tech SOP No. 024, "Recording Notes in Field Logbooks." Each sample will be collected into one Ziploc bag (one bag for each depth), labeled (site identification [ID], property ID, sample location number, depth interval, and date), and stored for later screening for lead and arsenic content with an XRF analyzer using the procedures defined in Section 7.0. The sampling device will be decontaminated with an Alconox detergent wash and rinsed with distilled water before sampling at each location.

Residential sampling locations, if deemed necessary, will be reviewed with the property owners or representatives in advance of subsurface activities to establish the location of private utilities and other obstructions. A one call service for utilities in Indiana (Indiana811) will also be contacted. The soil sample locations may need to be moved during the assessment based on various conditions, including but not limited to, gravel piles, excavated areas, utilities, and subsurface refusal encountered while sampling.

Drip zone samples will be collected from 0 to 6 inches bgs. At each of the other sample locations, a hand auger will be used to collect soil from 0 to 24 inches bgs. Aliquots will be collected from four depth intervals: 0 to 6 inches, 6 to 12 inches, 12 to 18 inches, and 18 to 24 inches bgs (EPA 2003). Aliquots from each depth interval will be composited with aliquots from the same depth interval from the other aliquot locations. Decontamination between sample locations will follow procedures outlined in Section 10.0 of this SAP.

Soil will be logged for lithology using the Unified Soil Classification System (USCS), noting any potential anthropogenic sources of lead (slag, sinter, ash, or similar materials). Significant observations (gross contamination) will be photographically documented. Soil samples will be collected in accordance with Tetra Tech SOP No. 005-2, "Soil Sampling," using hand auger sample methodology and collecting soil samples for non-volatile organic compound analyses. Aliquots from a given depth interval from each aliquot location will be placed in a plastic zip-tight baggie and thoroughly mixed for homogenization for each composite sample.

Each bag will be labeled to identify the boring location and associated property. The samples will be labeled with the site ID (Niagara LaSalle facility [NL], Property 01 [01], for example), sample description (Soil Boring [SB]), sample location number (01, 02, 03), sample depth interval, in inches bgs, sampling date (MMDDYY), and sample type, if the sample is a drip zone sample (DZ) or duplicate (D). Example soil sample IDs are provided in the table below.

Example Sample Nomenclature

Sample Type	Example Nomenclature
Niagara LaSalle facility soil sample	NL-SB-01-0006-080317
Residential soil sample	01-SB-01-0006-080317
Residential drip zone soil sample	01-SB-01-0006-080317-DZ
Duplicate soil sample	01-SB-01-0006-080317-D

Once samples have been placed in a labeled Ziploc bag, the soil in each plastic baggie will be homogenized by breaking up large chunks of soil and then mixing the soil within the bag. The plastic bag will then be screened with an XRF analyzer using the procedures described in Section 7.0.

The screening results will be used to select samples for off-site laboratory analysis. Anticipated laboratory sample totals are provided in Table 1 in Appendix B. Analytical methods, volumes, containers, preservation, holding times are provided in Table 2 in Appendix B.

Soil not selected for laboratory analysis will be returned to the hole from which it was removed, and the remaining open hole will be filled with clean topsoil.

7.0 XRF FIELD SCREENING AND SAMPLE SELECTION

The XRF screening will be conducted in accordance with Tetra Tech site specific SOP-029 "Field Portable XRF Analysis of Soil Samples." The bagged soil samples will be checked for moisture either visually or by use of a moisture meter. If no visible water is present in the soil, or the moisture content of the soil is below 20 percent, as measured by the moisture meter, then the sample will be screened for metals by EPA or START, using the hand-held XRF unit. If water is present, then the sample will be dried using a toaster oven on low heat until the water dissipates.

Given the heterogeneity of metals in most soils, the soil samples will be analyzed via the XRF unit three times per bag (at three different areas of the sample in the bag). Averages of the three values will be designated as the sample metals concentrations. All data will be stored digitally for upload into the EPA Scribe database.

All Niagara LaSalle facility soil samples and at least 20 percent of the residential soil samples will be selected for laboratory analysis of total metals. The samples will be selected for laboratory analysis to submit an approximately even distribution of sample intervals and sample locations with low (0 to 400 ppm), medium (400 to 800 ppm), and high (>800 ppm) XRF lead readings. Soil from the screened Ziploc bag will placed into a 4-ounce container for laboratory analysis for total metals analysis or into a 9-ounce container for TCLP metals. The remainder of the soil sampled will be stored at an EPA warehouse under custody seals until analytical results are received. START will also collect QA/QC samples as described in Section 9.0.

8.0 SAMPLE HANDLING

Sampling locations will be noted in the site logbook in accordance with Tetra Tech SOP No. 024, "Recording Notes in Field Logbooks." The samples collected will be labeled, packaged, and shipped in accordance with procedures outlined in Worksheets #26 and 27 of Tetra Tech's START Quality Assurance Project Plan (QAPP) (Tetra Tech 2016) and Tetra Tech SOP No.019-7, "Packaging and Shipping Samples."

9.0 QUALITY ASSURANCE/QUALITY CONTROL

QC samples will be collected to evaluate the field sampling and decontamination methods, and the overall reproducibility of the laboratory analytical results. Specifically, QC samples will be collected at the following frequencies:

- Field duplicate samples: 1 per 10 investigative samples
- Matrix spike/matrix spike duplicate samples: 1 per 20 investigative samples
- Equipment rinsate blank samples: 1 at the end of the sampling activities for the decontaminated hand auger

Each field duplicate sample will be collected from the same Ziploc bag that START used to collect the initial investigative soil sample. The field duplicate samples will be processed, stored, packaged, and analyzed by the same methods as the investigative samples. The sample nomenclature, specific to the QC samples, is described in Section 6.2 of this SAP.

The relative percent difference (RPD) between each field duplicate and investigative sample will be calculated by the START QA reviewer. RPDs greater than 70 percent (where detections are greater than the quantitation limit) will be summarized in a data validation report. START chemists will validate the laboratory results in accordance with EPA National Functional Guidelines (NFG) for Inorganic Superfund Methods Data Review (EPA 2017). Corrective actions may include resampling, reassessment of the laboratory's methods, or the addition of data qualifiers to the laboratory results.

Equipment rinsate blanks will be collected by slowly rinsing the decontaminated stainless hand auger with laboratory-grade deionized water while simultaneously collecting the used rinse water in a laboratory-provided container. Rinsate blank samples will be delivered to the designated laboratory under chain-of-custody for analysis for total metals. The table below lists equipment rinsate blank container and analytical requirements.

Matrix	Containers (Number, Size, and Type	Analytical Parameter(s)	Analytical Method	Preservation Requirement	Holding Time
Water	One 500- milliliter plastic bottle	Total metals analysis	SW846-6010, 7470A	Nitric Acid to pH <2; Cool to 4 degrees C	180 days (28 days for mercury)

EPA or START will be responsible for calibration of the XRF unit. XRF maintenance and calibration will follow the procedures provided in Table 3 of Appendix B. The Tetra Tech project manager, Rachel Houle, will be responsible for ensuring that sample quality and integrity are maintained and that sample labeling and documentation procedures are in accordance with the QAPP.

10.0 DECONTAMINATION

Personal protective equipment (PPE) will be double-bagged and disposed of as dry, industrial waste.

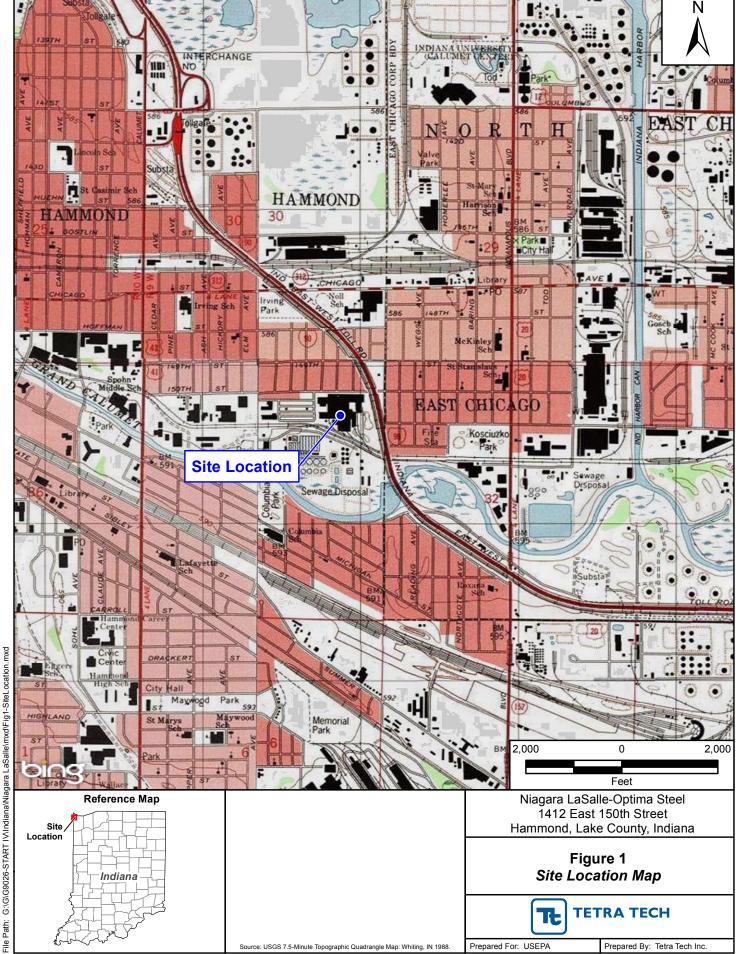
Non-disposable equipment (such as the hand auger) will be decontaminated between sampling locations following the procedures in Tetra Tech SOP No. 002, "General Equipment Decontamination."

Decontamination water use will be kept to a minimum, and typically 5 to 10 gallons of rinsate water is generated. The rinsate water will be disposed of on site by evaporation over a hard surface.

11.0 REFERENCES

- Tetra Tech Inc. (Tetra Tech). 2016. Superfund Technical Assessment and Response Team (START IV), Revision 1, EPA Region 5, Contract No. EP-S5-EP-01, Quality Assurance Project Plan (QAPP). June.
- U.S. Environmental Protection Agency (EPA). 2003. Superfund Lead-Contaminated Residential Sites Handbook. Environmental Protection Agency Lead Sites Workgroup. OSWER 9285.7-50.
- EPA. 2017. National Functional Guidelines for Inorganic Superfund Methods Data Review. Office of Superfund Remediation and Technology Innovation. OLEM 9355.0-135. January.

APPENDIX A Figures (2)



TDD No.: S05-0001-1707-004

Date Saved: 7/27/2017 EPA Contract No.: EP-S5-13-01

Coordinate System: GCS WGS 1984 Datum: WGS 1984 Units: Degree

APPENDIX B Tables (3)

TABLE 1: SOIL SAMPLE SUMMARY

Analytical Parameter	Analytical Method	Number of Soil Samples ¹	Number of Field Duplicates	Number of MS/MSDs	Number of Blanks (Rinsate)	Total Number of Samples to Laboratory ²
Total Metals	SW846-6010C, 7471B	Unknown number of XRF Samples and Laboratory Samples	1 duplicate every 10 samples	1 MS/MSD for every 20 samples	1 per sampling event	TBD
TCLP Metals	SW846-1311, 6010C, 7470A	Unknown number of Laboratory Samples	1 submitted to lab	NA	NA	TBD

Notes:

The number of samples will be determined after the site visit on August 2; for subsequent residential sampling, the number of

samples depends on property access and size.

2 Total number of samples to the laboratory does not include MS/MSD samples

MS/MSD Matrix Spike/Matrix Spike Duplicate

XRF X-ray fluorescence

TABLE 2: ANALYTICAL METHODS

Matrix	Parameter	Analytical	Volumes and Containers	Preservation	Holding Time ¹
		Method			
Soil	Total Metals	SW846-6010C,	One 4-oz glass jar with	Cool to 4 $^{\circ}$ C \pm 2	180 days for all
		7471B	polyethylene-lined cap	°C immediately	metals (28 days
				after collection	for mercury)
Soil	TCLP Metals	SW846-1311,	One 9-oz glass jar with	Cool to 4 $^{\circ}$ C \pm 2	180 days for all
		6010C, 7470A	polyethylene-lined cap	°C immediately	metals (28 days
				after collection	for mercury)

Notes:

Holding time is measured from the time of sample collection to the time of sample extraction and analysis

TCLP Toxicity Characteristic Leaching Procedure

TABLE 3: XRF CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

Field	Calibration	Maintenance	Testing/Inspection	Frequency	Acceptance	Corrective	Responsible
Equipment	Activity	Activity	Activity		Criteria	Action	Person
XRF	Per manufacturer's instruction	Check calibration date in internal computer system; cycle battery when discharge rate becomes frequent	Calibration check, test battery, inspect cords and wire, ensure Mylar cover is intact	Daily before first measurement and after every 20 samples thereafter	Standard results must be within ±30% of true value	Repeat calibration; service if calibration issues persist	Equipment vendor (EPA)

Notes:

XRF X-ray fluorescence

Appendix C

Tetra Tech Standard Operating Procedures

SOP 002-3 (General Equipment Decontamination)

SOP 005-2 (Soil Sampling)

SOP 019-7 (Packaging and Shipping Samples)

SOP 024-2 (Recording Notes in Field Logbooks)

Site Specific SOP 029 (Field Portable X-Ray Fluorescence Analysis of Soil Samples)

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

GENERAL EQUIPMENT DECONTAMINATION

SOP NO. 002

REVISION NO. 3

Last Reviewed: June 2009

•		
Kresing	6-19-09	
Quality Assurance Approved	Date	

Revision No. 3, June 2009 Last Reviewed: June 2009

1.0 BACKGROUND

All nondisposable field equipment must be decontaminated before and after each use at each sampling location to obtain representative samples and to reduce the possibility of cross-contamination.

1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for decontaminating equipment in the field.

1.2 SCOPE

This SOP applies to decontaminating general nondisposable field equipment. To prevent contamination of samples, all sampling equipment must be thoroughly cleaned prior to each use.

1.3 **DEFINITIONS**

Alconox: Nonphosphate soap, obtained in powder detergent form and dissolved in water

Liquinox: Nonphosphate soap, obtained in liquid form for mixing with water

1.4 REFERENCES

- U.S. Environmental Protection Agency (EPA). 1992a. "Guide to Management of Investigation-Derived Wastes." Office of Solid Waste and Emergency Response. Washington D.C. EPA 9345.3-03FS. January.
- EPA. 1992b. "RCRA Ground-Water Monitoring: Draft Technical Guidance." Office of Solid Waste. Washington, DC. EPA/530-R-93-001. November.
- EPA. 1994. "Sampling Equipment Decontamination." Environmental Response Team SOP #2006 (Rev. #0.0, 08/11/94). http://www.ert.org/mainContent.asp?section=Products&subsection=List

1.5 REQUIREMENTS AND RESOURCES

The equipment required to conduct decontamination is as follows:

- · Scrub brushes
- · Large wash tubs or buckets
- · Squirt bottles
- · Alconox or Liquinox
- · Tap water
- Distilled water
- · Plastic sheeting
- · Aluminum foil
- · Methanol or hexane
- · Isopropanol (pesticide grade)
- · Dilute (0.1 N) nitric acid

2.0 PROCEDURE

The procedures below discuss decontamination of personal protective equipment (PPE), drilling and monitoring well installation equipment, borehole soil sampling equipment, water level measurement equipment, general sampling equipment, and groundwater sampling equipment.

2.1 PERSONAL PROTECTIVE EQUIPMENT DECONTAMINATION

Personnel working in the field are required to follow specific procedures for decontamination prior to leaving the work area so that contamination is not spread off site or to clean areas. All used disposable protective clothing, such as Tyvek coveralls, gloves, and booties, will be containerized for later disposal. Decontamination water will be containerized in 55-gallon drums (refer to Section 3.0).

Personnel decontamination procedures will be as follows:

- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of personal protection as was used for sampling.

- 3. Wash neoprene boots (or neoprene boots with disposable booties) with Liquinox or Alconox solution and rinse with clean water. Remove booties and retain boots for subsequent reuse.
- 4. Wash outer gloves in Liquinox or Alconox solution and rinse in clean water. Remove outer gloves and place into plastic bag for disposal.
- 5. Remove Tyvek or coveralls. Containerize Tyvek for disposal and place coveralls in plastic bag for reuse.
- 6. Remove air purifying respirator (APR), if used, and place the spent filters into a plastic bag for disposal. Filters should be changed daily or sooner depending on use and application. Place respirator into a separate plastic bag after cleaning and disinfecting.
- 7. Remove disposable gloves and place them in plastic bag for disposal.
- 8. Thoroughly wash hands and face in clean water and soap.

2.2 DRILLING AND MONITORING WELL INSTALLATION EQUIPMENT **DECONTAMINATION**

All drilling equipment should be decontaminated at a designated location on site before drilling operations begin, between borings, and at completion of the project. Decontamination may be conducted on a temporary decontamination pad constructed at satellite locations within the site area in support of temporary work areas. The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Decontamination pads may be constructed of concrete, wood, or plastic sheeting, depending on the site-specific needs and plans. Wash waters and contaminated soil generated during decontamination activities should be considered contaminated and thus, should be collected and containerized for proper disposal.

Monitoring well casing, screens, and fittings are assumed to be delivered to the site in a clean condition. However, they should be steam cleaned and placed on polyethylene sheeting on-site prior to placement downhole. The drilling subcontractor will typically furnish the steam cleaner and water.

The drilling auger, bits, drill pipe, any portion of drill rig that is over the borehole, temporary casing, surface casing, and other equipment used in or near the borehole should be decontaminated by the drilling subcontractor as follows:

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- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of personal protection as was used for sampling.
- 3. Remove loose soil using shovels, scrapers, wire brush, etc.
- 4. Steam clean or pressure wash to remove all visible dirt.
- 5. If equipment has directly or indirectly contacted contaminated media and is known or suspected of being contaminated with oil, grease, polynuclear aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB), or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol.
- 6. To the extent possible, allow components to air dry.
- 7. Wrap or cover equipment in clear plastic until it is time to be used.
- 8. All wastewater from decontamination procedures should be containerized.

2.3 BOREHOLE SOIL SAMPLING DOWNHOLE EQUIPMENT DECONTAMINATION

All soil sampling downhole equipment should be decontaminated before use and after each sample as follows:

- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of personal protection as was used for sampling.
- 3. Prior to sampling, scrub the split-barrel sampler and sampling tools in a wash bucket or tub using a stiff, long bristle brush and Liquinox or Alconox solution.
- 4. After sampling, steam clean the sampling equipment over the rinsate tub and allow to air dry.
- 5. Place cleaned equipment in a clean area on plastic sheeting and wrap with aluminum foil.
- 6. Containerize all water and rinsate; disposable single-use sampling equipment should also be containerized.
- 7. Decontaminate all equipment placed down the hole as described for drilling equipment.

2.4 WATER LEVEL MEASUREMENT EQUIPMENT DECONTAMINATION

Field personnel should decontaminate the well sounder and interface probe before inserting and after removing them from each well. The following decontamination procedures should be used:

- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of personal protection as was used for sampling.
- 3. Wipe the tape and probe with a disposable Alconox- or Liquinox-impregnated cloth or paper towel.
- 4. If immiscible layers are encountered, the interface probe may require steam cleaning or washing with pesticide-grade isopropanol.
- 5. Rinse with deionized water.

2.5 GENERAL SAMPLING EQUIPMENT DECONTAMINATION

All nondisposable sampling equipment should be decontaminated using the following procedures:

- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of personal protection as was used for sampling.
- 3. To decontaminate a piece of equipment, use an Alconox wash; a tap water wash; a solvent (isopropanol, methanol, or hexane) rinse, if applicable, or dilute (0.1 N) nitric acid rinse, if applicable; a distilled water rinse; and air drying. Use a solvent (isopropanol, methanol, or hexane) rinse for grossly contaminated equipment (for example, equipment that is not readily cleaned by the Alconox wash). The dilute nitric acid rinse may be used if metals are the analyte of concern.
- 4. Place cleaned equipment in a clean area on plastic sheeting and wrap with aluminum foil.
- 5. Containerize all water and rinsate.

2.6 GROUNDWATER SAMPLING EQUIPMENT

The following procedures are to be employed for the decontamination of equipment used for groundwater sampling. Decontamination is not necessary when using disposable (single-use) pump tubing or bailers. Bailer and downhole pumps and tubing decontamination procedures are described in the following sections.

2.6.1 Bailers

- Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of personal protection as was used for sampling.
- 3. Evacuate any purge water in the bailer.
- 4. Scrub using soap and water and/or steam clean the outside of the bailer.
- 5. Insert the bailer into a clean container of soapy water. Thoroughly rinse the interior of the bailer with the soapy water. If possible, scrub the inside of the bailer with a scrub brush.
- 6. Remove the bailer from the container of soapy water.
- 7. Rinse the interior and exterior of the bailer using tap water.
- 8. If groundwater contains or is suspected to contain oil, grease, PAH, PCB, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol.
- 9. Rinse the bailer interior and exterior with deionized water to rinse off the tap water and solvent residue, as applicable.
- 10. Drain residual deionized water to the extent possible.
- 11. Allow components to air dry.
- 12. Wrap the bailer in aluminum foil or a clean plastic bag for storage.
- 13. Containerize the decontamination wash waters for proper disposal.

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2.6.2 Downhole Pumps and Tubing

- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of personal protection as was used for sampling.
- 3. Evacuate any purge water in the pump and tubing.
- 4. Scrub using soap and water and/or steam clean the outside of the pump and, if applicable, the pump tubing.
- 5. Insert the pump and tubing into a clean container of soapy water. Pump/run a sufficient amount of soapy water to flush out any residual well water. After the pump and tubing are flushed, circulate soapy water through the pump and tubing to ensure that the internal components are thoroughly flushed.
- 6. Remove the pump and tubing from the container.
- 7. Rinse external pump components using tap water.
- 8. Insert the pump and tubing into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).
- 9. If groundwater contains or is suspected to contain oil, grease, PAH, PCB, or other hard to remove organic materials, rinse the pump and tubing with pesticide-grade isopropanol.
- 10. Rinse the pump and tubing with deionized water to flush out the tap water and solvent residue, as applicable.
- 11. Drain residual deionized water to the extent possible.
- 12. Allow components to air dry.
- 13. For submersible bladder pumps, disassemble the pump and wash the internal components with soap and water, rinse with tap water, isopropanol (if necessary), and deionized water, and allow to air dry.
- 14. Wrap pump and tubing in aluminum foil or a clean plastic bag for storage.
- 15. Containerize the decontamination wash waters for proper disposal.

3.0 INVESTIGATION-DERIVED WASTE

Investigation-derived waste (IDW) can include disposable single-use PPE and sampling equipment, soil cuttings, and decontamination wash waters and sediments. Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage will be provided in project-specific documents, or separate direction will be provided by the project manager. The following guidelines are provided for general use:

- 1. Assume that all IDW generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.
- 2. Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.
- 3. Label IDW storage containers with the facility name and address, date, contents, company generating the waste, and an emergency contact name and phone number.
- 4. Temporarily store the IDW in a protected area that provides access to the containers and allows for spill/leak monitoring, sampling of containers, and removal following determination of the disposal method.

SOP APPROVAL FORM

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

SOIL SAMPLING

SOP NO. 005

REVISION NO. 2

Last Reviewed: June 2009

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Quality Assurance Approved
Date

1.0 **BACKGROUND**

Soil sampling is conducted for three main reasons: for laboratory chemical analysis, laboratory physical analysis, or visual classification and field screening. These three sampling objectives can be achieved separately or in combination with each other. Sampling locations are typically chosen to provide chemical, physical, or visual information in both the horizontal and vertical directions. A sampling and analysis plan is used to outline sampling methods and provide preliminary rationale for sampling locations. Sampling locations may be adjusted in the field based on the screening methods being used and the physical features of the area.

1.1 **PURPOSE**

Soil sampling is conducted to determine the chemical, physical, and visual characteristics of surface and subsurface soils.

1.2 **SCOPE**

This standard operating procedure (SOP) describes procedures for soil sampling in different areas using various implements. It includes procedures for test pit, surface soil, and subsurface soil sampling, and describes ten soil sampling devices.

1.3 **DEFINITIONS**

Hand auger: Instrument attached to the bottom of a length of pipe that has a crossarm or "T" handle at the top. The auger can be closed-spiral or open-spiral.

Bucket auger: A type of auger that consists of a cylindrical bucket 10 to 72 inches in diameter with teeth arranged at the bottom.

Core sampler: Thin-wall cylindrical metal tube with diameter of 0.5 to 3 inches, a tapered nosepiece, a "T" handle to facilitate sampler deployment and retrieval, and a check valve (flutter valve) in the headpiece.

EnCoreTM sampler: A disposable volumetric sampling device. It comes in sample sizes of 5 and 25 grams. It is a hermetically sealed, single-use soil sampler made from a high-tech, inert polymer. EnCoreTM samplers are used to collect soil samples with zero headspace, as required for volatile organic compound analysis. Each sample is collected using a reuseable "T" handle.

Spatulas or Spoons: Stainless steel or disposable instruments for collecting loose unconsolidated material.

Trier: Tube cut in half lengthwise with a sharpened tip that allows for collection of sticky solids or loosening of cohesive soils.

Trowel: Metal or disposable tool with a scooped blade 4 to 8 inches long and 2 to 3 inches wide with a handle.

Split-Spoon (or Split-Barrel) Sampler: Thick-walled steel tube that is split lengthwise. A cutting shoe is attached to the lower end; the upper end contains a check valve and is connected to drill rods.

Thin-Wall Tube Sampler: Steel tube (1 to 3 millimeters thick) with a tapered bottom edge for cutting. The upper end is fastened to a check valve that is attached to drill rods.

Volatile Organics Analysis (VOA) Plunger: Disposable, plastic, single-use soil sample collection device for volatile organic compound sample collection.

1.4 REFERENCES

- U.S. Environmental Protection Agency. (EPA) 1984. "Soil Sampling Quality Assurance Users Guide." EPA 600/4-84-043.
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1.5 REQUIREMENTS AND RESOURCES

Soil sampling requires the use of one or more of the following types of equipment:

- Spoons and spatulas
- Trowel
- Shovel or spade
- Trier
- Core sampler
- EnCoreTM sampler
- VOA Plunger
- Hand auger
- Bucket auger
- Split-spoon
- Thin-wall tube

In addition, the following equipment is also needed for various methods:

- Sample containers, labels, and chain-of-custody forms
- Logbook
- Tape for measuring recovery
- Soil classification information
- Wax or caps for sealing ends of thin-wall tube
- "T" Handles
- Plastic sheeting
- Decontamination equipment
- Drilling equipment
- Backhoe
- Health and safety equipment

2.0 SOIL SAMPLING PROCEDURES

This SOP presents procedures for conducting test pit, surface soil, and subsurface soil sampling. The site sampling plan will specify which of the following procedures will be used.

Soil samples for chemical analysis should be collected in the following order: (1) volatile organics, (2) semivolatile organics, and (3) metals. Once the chemical samples have been containerized, samples for physical analyses can be containerized. Typical physical analyses conducted include (1) grain size distribution, (2) moisture content, (3) saturated permeability, (4) unsaturated permeability, and (5) Atterberg limits. Additionally, visual descriptions of samples, using the Unified Soil Classification System (USCS), should be recorded. Field tests such as head space analyses can also be conducted.

Soil samples for chemical analyses can be collected either as grab samples or composite samples. A grab sample is collected from a discrete location or depth. A composite sample consists of soil combined from more than one discrete location. Typically, composite samples consist of soil obtained from several locations and homogenized in a stainless steel or Teflon® pan, tray, or baggie. Refer to the site-specific Quality Assurance Project Plan (QAPP) for methodology for composite sample collection. Samples for volatile organics analysis should not be composited.

All soil samples collected should be packaged and shipped to the laboratories in accordance with SOP 019. All nondedicated or nondisposable equipment used for soil sampling should be decontaminated between sampling locations in accordance with SOP 002.

2.1 SOIL SAMPLE COLLECTION PROCEDURES

Soil samples can be collected as discrete samples for volatile organic compound (VOC) analysis using specialized equipment for preservation in the laboratory or in the field. Soil samples collected for non-VOC analysis can be collected as either grab or composite samples using standard equipment.

2.1.1 Procedure for Preserving and Collecting Soil Samples for VOC analysis

Samples collected for VOC analysis using traditional methods, such as collection in a jar with no preservation, are shown to yield nonrepresentative samples due to loss of VOCs. To prevent such losses, preservation with methanol or sodium bisulfite may be used to minimize volatilization and biodegradation. This preservation may be performed in the laboratory or in the field, depending on the sample collection methodology used. The specific sampling methodology will be specified in the projectspecific QAPP or work plan.

Soil samples to be preserved in the laboratory are collected using SW-846 Method 5035. For samples preserved in the field, laboratories may perform low-level analysis (sodium bisulfate preservation) or high- to medium-level analyses (methanol preservation), depending on the project-specific QAPP.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

2.1.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for VOC analysis that are to be preserved at the laboratory shall be obtained using a hand-operated, hermetically sealed sample vial such as an EnCoreTM sampler. Each sample shall be obtained using a reusable sampling handle ("T" handle) that can be provided with the EnCoreTM sampler when requested and purchased. Collect the soil sample in the following manner for each EnCoreTM sampler.

The EnCoreTM sampler is loaded into the "T" handle with the plunger fully depressed. Press the "T" handle into the soil to be sampled. The plunger will be forced upward as the cavity fills with soil. When the sampler is full, using the "T" handle, rotate the plunger and lock it into place. If the plunger does not lock, then it is not filled with soil. Soft soil may require several plunges or forcing soil against a hard surface such as a decontaminated sample trowel to ensure headspace has been eliminated. Remove soil from the outside of the sampler so a tight seal can be made between the sample cap and the O-ring. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler. Remove any surface soil from outside of the sampler and place in the foil bag provided with the sampler. Label the bag with sample location information. Typically, collect three EnCoreTM samplers per sample location. Decontaminate the "T" handle between sample locations.

Using the EnCoreTM sampler eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each EnCoreTM sampler. After the EnCoreTM samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours. The samples must be preserved by the laboratory within 48 hours of collection.

2.1.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analysis using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods. If samples effervesce when placed in preservative, it is necessary to collect a sample unpreserved, in deionized water. In addition, an unpreserved sample for determination of moisture content must also be collected when collecting soil samples to be preserved in the field.

Methanol Preservation (High to Medium Level). Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40- to 60-milliliter (mL) glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade 3 methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable VOA plunger. The outside diameter of the coring device must be smaller than the inside of the sample bottle neck. To collect the sample, pull the plunger back to the required location, insert it into the soil to be sampled, push the coring device into the soil, extrude the soil sample into the methanol-preserved sample bottle, and cap the bottle tightly. Swirl the sample (do not shake) in the methanol to break up the soil such that all of the soil is covered with methanol. Place the sample on ice immediately.

Sodium Bisulfate Preservation (Low Level). Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be field-preserved using sodium bisulfate are collected using the same procedures described for methanol preservation.

2.1.2 Procedure for Collecting Soil Samples for Non-VOC Analyses

Samples collected for non-VOC analyses may be collected as either grab or composite samples as follows. Using a sampling device, transfer a portion of soil to be sampled to a stainless steel bowl, disposable inert plastic tray, or baggie. Remove roots, vegetation, sticks, and stones larger than the size of pea gravel. Thoroughly mix the soil to obtain as uniform a texture and color as practicable. Transfer the mixed soil to the appropriate sample containers and close the containers. Place the sample containers immediately on ice.

2.2 TEST PIT AND TRENCH SOIL SAMPLING

Test pit and trench soil sampling is conducted when a complete soil profile is required or as a means of locating visually detectable contamination. This type of sampling provides a detailed description of the soil profile and allows for multiple samples to be collected from specific soil horizons. Prior to conducting any test pit or trench excavation with a backhoe, the sampling team should ensure that the sampling area is clear of utility lines, subsurface pipes, and poles.

A test pit or trench is excavated by incrementally removing soil material with a backhoe bucket. The excavated soil is placed on plastic sheeting well away from the edge of the test pit. A test pit should not be excavated to depths greater than 4 feet unless its walls are properly sloped or stabilized. No personnel shall enter any test pit or trench excavation over 4 feet deep; such action would constitute confined space entry and must conform with Occupational Safety and Health Administration (OSHA) regulations at Title 29 of the *Code of Federal Regulations* § 1910.

Personnel entering the test pit may be exposed to toxic or explosive gases and oxygen deficient environments. Air monitoring is required before entering the test pit, and the use of appropriate respiratory gear and protective clothing is mandatory. At least two persons must be present at the test pit before sampling personnel may enter the excavation and begin soil sampling. Refer to project-specific Health and Safety Plans for required safety procedures for excavations.

Soil samples can also be obtained directly from the backhoe bucket or from the excavated material after it has been removed and deposited on plastic sheeting. The sampling personnel shall direct the backhoe excavator to obtain material from the selected depth and location within the excavation. The backhoe

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operator shall set the backhoe bucket on the ground in a designated location, at a sufficient distance from the excavation to allow the sampler safe access to the bucket. The backhoe operator shall disengage the controls and signal to the sampler that it is safe to approach the bucket. The soil sample shall then be collected from the center of the backhoe bucket to reduce the potential for cross-contamination of the sample.

Test pits are not practical for sampling at depths greater than 15 feet. If soil samples are required from depths greater than 15 feet, samples should be obtained using test borings instead of test pits. Test pits are also usually limited to a few feet below the water table. In some cases, a pumping system may be required to control the water level within the pits.

Access to open test pits should be restricted by the use of flagging, tape, or fencing. If a fence is used, it should be erected at least 6 feet from the perimeter of the test pit. The test pit should be backfilled as soon as possible after sampling is completed.

Various equipment may be used to collect soil samples from the walls or bottom of a test pit. A hand auger, bucket auger, or core sampler can be used to obtain samples from various depths. A trier, trowel, EnCoreTM sampler, VOA plunger, or spoon can be used to obtain samples from the walls or pit bottom surface.

2.3 SURFACE SOIL SAMPLING

The surface soil sampling equipment presented in this SOP is best suited for sampling to depths of 0 to 6 feet below ground surface (bgs). The sample depth, sample analyses, soil type, and soil moisture will also dictate the most suitable sampling equipment. Prior to sample collection, the sampling locations should be cleared of any surface debris such as twigs, rocks, and litter. The following table presents various surface soil sampling equipment and their effective depth ranges, operating means (manual or power), and sample types collected (disturbed or undisturbed).

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Sampling Equipment	Effective Depth Range (feet bgs)	Operating Means	Sample Type
Hand Auger	0 to 6	Manual	Disturbed
Bucket Auger	0 to 4	Power	Disturbed
Core Sampler	0 to 4	Manual or Power	Undisturbed
EnCore TM Sampler	Not Applicable	Manual	Disturbed
Spoon/Spatula	0 to 0.5	Manual	Disturbed
Trowel	0 to 1	Manual	Disturbed
VOA Plunger	Not Applicable	Manual	Disturbed

The procedures for using these various types of sampling equipment are discussed below.

2.3.1 **Hand Auger**

A hand auger equipped with extensions and a "T" handle is used to obtain samples from depths of up to 6 feet bgs. If necessary, a shovel may be used to excavate the topsoil to reach the desired subsoil level. If topsoil is removed, its thickness should be recorded. Samples obtained using a hand auger are disturbed in their collection; determining the exact depth at which samples are obtained is difficult.

The hand auger is screwed into the soil at an angle of 45 to 90 degrees from horizontal. When the entire auger blade has penetrated soil, the auger is removed from the soil by lifting it straight up without turning it, if possible. If the desired sampling depth has not been reached, the soil is removed from the auger and deposited onto plastic sheeting. This procedure is repeated until the desired depth is reached and the soil sample is obtained. The auger is then removed from the boring and the soil sample is collected directly from the auger into an appropriate sample container.

2.3.2 **Bucket Auger**

A bucket auger, equipped similarly as the hand auger, is used to obtain disturbed samples from depths of

up to 4 feet bgs. A bucket auger should be used when sampling stony or dense soil that prohibits the use

of a hand-operated core or screw auger. A bucket auger with closed blades is used in soil that cannot

generally be penetrated or retrieved by a core sampler.

The bucket auger is rotated while downward pressure is exerted until the bucket is full. The bucket is

then removed from the boring, the collected soil is placed on plastic sheeting, and this procedure is

repeated until the appropriate depth is reached and a sample is obtained. The bucket is then removed

from the boring and the soil sample is transferred from the bucket to an appropriate sample container.

2.3.3 **Core Sampler**

A hand-operated core sampler (Figure 1), similarly equipped as the hand auger, is used to obtain samples

from depths of up to 4 feet bgs in uncompacted soil. The core sampler is capable of retrieving

undisturbed soil samples and is appropriate when low concentrations of metals or organics are of concern.

The core sampler should be constructed of stainless steel. A polypropylene core sampler is generally not

suitable for sampling dense soils or sampling at greater depths.

The core sampler is pressed into the soil at an angle of 45 to 90 degrees from horizontal and is rotated

when the desired depth is reached. The core is then removed, and the sample is placed into an appropriate

sample container.

2.3.4 **Shovel**

A shovel may be used to obtain large quantities of soil that are not readily obtained with a trowel. A

shovel is used when soil samples from depths of up to 6 feet bgs are to be collected by hand excavation; a

tiling spade (sharpshooter) is recommended for excavation and sampling. A standard steel shovel may be

used for excavation; either a stainless steel or polypropylene shovel may be used for sampling. Soil

excavated from above the desired sampling depth should be stockpiled on plastic sheeting. Soil samples

should be collected from the shovel and placed into the sample container using a stainless-steel scoop,

plastic spoon, or other appropriate tool.

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A trier (Figure 2) is used to sample soil from depths up to 1 foot bgs. A trier should be made of stainless steel or polypropylene. A chrome-plated steel trier may be suitable when samples are to be analyzed for organics and heavy metal content is not a concern.

Samples are obtained by inserting the trier into soil at an angle of up to 45 degrees from horizontal. The trier is rotated to cut a core and is then pulled from the soil being sampled. The sample is then transferred to an appropriate sample container.

2.3.6 **Trowel**

A trowel is used to obtain surface soil samples that do not require excavation beyond a depth of 1 foot. A trowel may also be used to collect soil subsamples from profiles exposed in test pits. Use of a trowel is practical when sample volumes of approximately 1 pint (0.5 liter) or less are to be obtained. Excess soil should be placed on plastic sheeting until sampling is completed. A trowel should be made of stainless steel or galvanized steel. It can be purchased from a hardware or garden store. Soil samples to be analyzed for organics should be collected using a stainless steel trowel. Samples may be placed directly from the trowel into sample containers.

2.4 SUBSURFACE SOIL SAMPLING

Subsurface soil sampling is accomplished in conjunction with borehole drilling, for soil sampling from depths greater than approximately 6 feet bgs. Subsurface soil sampling is frequently coupled with exploratory boreholes or monitoring well installation.

Subsurface soil sampling may be conducted using a drilling rig, power auger, or direct-push technology (DPT). Selection of sampling equipment depends upon geologic conditions and the scope of the sampling program. Two types of samplers used with machine-driven augers—the split-spoon sampler and the thin-wall tube sampler—are discussed below. All sampling tools should be cleaned before and after each use in accordance with SOP No. 002 (General Equipment Decontamination). Both the splitspoon sampler and the thin-wall tube sampler can be used to collect undisturbed samples from

unconsolidated soils. The procedures for using the split-spoon and thin-wall tube samplers are presented below.

2.4.1 **Split-Spoon Sampler**

Split-spoon samplers are available in a variety of types and sizes. Site conditions and project needs, such as large sample volume for multiple analyses, determine the specific type of split-spoon sampler to be used. Figure 3 shows a generic split-spoon sampler.

The split-spoon sampler is advanced into the undisturbed soil beneath the bottom of the casing or borehole using a weighted hammer and a drill rod. The relationship between hammer weight, hammer drop, and number of blows required to advance the split-spoon sampler in 6-inch increments indicates the density or consistency of the subsurface soil. After the split-spoon sampler has been driven to its intended depth, it should be removed carefully to avoid loss of sample material. In noncohesive or saturated soil, a catcher or basket should be used to help retain the sample.

After the split-spoon sampler is removed from the casing, it is detached from the drill rod and opened. If VOA samples are to be collected, EnCoreTM samplers or VOA plungers should be filled with soil taken directly from the split-spoon sampler. Samples for other specific chemical analyses should be taken as soon as the VOA sample has been collected. The remainder of the recovered soil can then be used for visual classification of the sample and containerized for physical analysis. The entire sample (except for the top several inches of possibly disturbed material) is retained for analysis or disposal.

2.4.2 **Thin-Wall Tube Sampler**

A thin-wall tube sampler, sometimes called the Shelby tube (Figure 4), is used to collect soil samples for geophysical analysis. Tube samplers are best suited for collecting cohesive soils such as clays and silts. The tube sampler may be pressed or driven into soil inside a hollow-stem auger flight, wash bore casing, or uncased borehole. The tube sampler is pressed into the soil, without rotation, to the desired depth or until refusal. If the tube cannot be advanced by pushing, it may be necessary to drive it into the soil without rotation using a hammer and drill rod. The tube sampler is then rotated to collect the sample from the soil and removed from the borehole.

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After removal of the tube sampler from the drilling equipment, the tube sampler should be inspected for adequate sample recovery. The sampling procedure should be repeated until an adequate soil core is obtained (if sample material can be retained by the tube sampler). The soil core obtained should be documented in the logbook. Any disturbed soil is removed from each end of the tube sampler. If chemical analysis is required, VOA samples must be collected immediately after the tube sampler is withdrawn. EnCoreTM samplers or VOA plungers should be filled with soil taken directly from the tube sampler. Before use, and during storage and transport, the tube sampler should be capped with a nonreactive material. For physical sampling parameters, the tube is sealed using plastic caps. The top and bottom of the tube sampler should be labeled and the tube sampler should be stored accordingly.

2.4.3 Direct-Push Technology Sampler

Geoprobe systems utilize DPT. In many cases, DPT is less expensive and faster than collecting soil samples with a standard drilling rig. In addition, the use of DPT causes minimal disturbance to the ground surface and generates little to no soil cuttings. DPT uses acetate or clear polyvinyl chloride (PVC) sleeves for collecting soil samples. Use of the Geoprobe system is described in SOP No. 054.

Upon retrieval of the sampling rod from the ground, the sample sleeve is extruded from the sampling rod. The sleeve is sliced lengthwise twice, to open the sleeve. Soil samples can be collected directly from the opened sleeve. If VOA samples are to be collected, EnCoreTM samplers or VOA plungers should be filled with soil taken directly from the opened DPT sampler. Samples for other specific chemical analyses should be taken after the VOA sample has been collected. The remainder of the recovered soil can then be used for visual classification of the sample and containerized for physical analysis. The entire sample is retained for analysis or disposal.

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FIGURE 1
HAND-OPERATED CORE SAMPLER

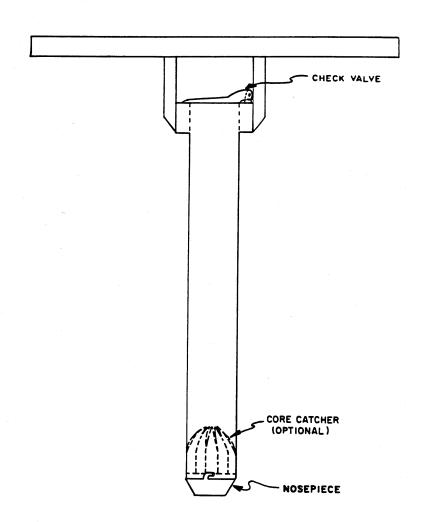
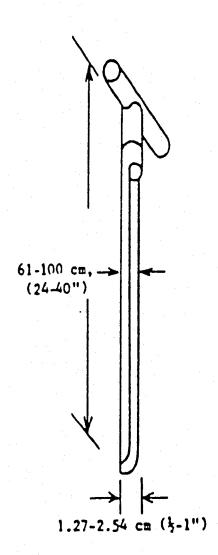


FIGURE 2

TRIER

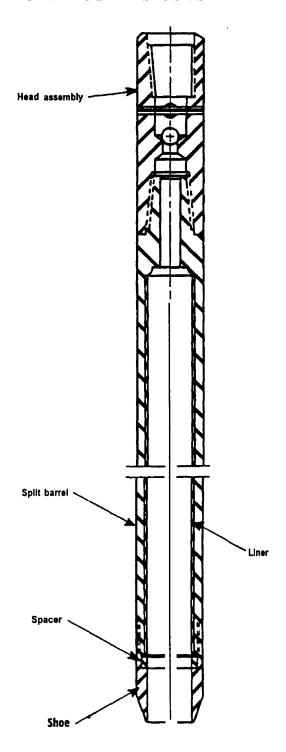
TRIER



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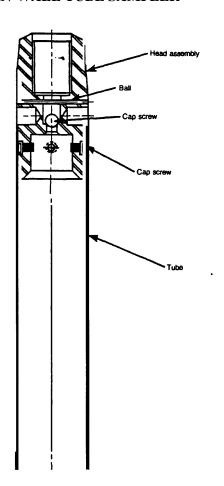
FIGURE 3
GENERIC SPLIT-SPOON SAMPLER



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FIGURE 4
THIN-WALL TUBE SAMPLER



SOP APPROVAL FORM

TETRA TECH, INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

PACKAGING AND SHIPPING SAMPLES

SOP NO. 019

REVISION NO. 7

Last Reviewed: November 2014

Carla J Bunk	
	November 24, 2014
Ouality Assurance Approved	Date

Title: Packaging and Shipping Samples

Revision No. 7, November 2014 Last Reviewed: November 2014

1.0 BACKGROUND

In any sampling program, the integrity of a sample must be ensured from its point of collection to its final disposition. This standard operating procedure (SOP) describes procedures for packaging and shipping samples. Steps in the procedures should be followed to ensure sample integrity and to protect the welfare of persons involved in shipping and receiving samples.

1.1 PURPOSE

This SOP establishes the requirements and procedures for packaging and shipping samples. It has been prepared in accordance with the U.S. Environmental Protection Agency (EPA) "Contract Laboratory Program Guidance for Field Samplers." Procedures described in this SOP should be followed for all routine sample packaging and shipping. If procedures are to be modified for particular contract- or laboratory-specific requirements, modified procedures should be clearly described in site-specific plans such as work plans, field sampling plans (FSPs), or quality assurance project plans (QAPPs). Deviations from the procedures in this SOP must be documented in a field logbook. This SOP assumes that samples are already in the appropriate sample jars and that the sample jars are labeled.

This SOP does not cover the packaging and shipment of Dangerous Goods or Hazardous Materials.

The shipment of Dangerous Goods (by air) and Hazardous Materials (by ground) requires specialized training. If you have NOT received this training in the last two years, you are NOT qualified to package or ship these materials and may be personally liable for any damages or fines. Contact one of Tetra Tech's shipping experts for assistance. Instructions to access the training course, shipping experts and health and safety (H&S) contacts, and general information on packaging and shipping hazardous substances and dangerous goods can be obtained by checking the links provided in Section 1.4 (References).

1.2 SCOPE

This SOP applies to packaging and shipping of environmental and nonhazardous samples. This SOP does not address shipping dangerous goods or hazardous materials.

1.3 **DEFINITIONS**

Airbill: An airbill is a shipping form (such as a FedEx shipping form) acquired from the commercial shipper and is used to document shipment of the samples from the sampler to the designated analytical laboratory (see Figure 1).

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Custody-of-Custody form: A chain-of-custody form is used to document the transfer of custody of samples from the field to the designated analytical laboratory (see Figure 2). The chain-of-custody form is critical to the chain-of-custody process and is used to identify the samples in each shipping container to be shipped or delivered to the laboratory for chemical or physical (geotechnical) analysis (see Figure 3).

Custody seal: A custody seal is a tape-like seal and is used to indicate that samples are intact and have not been disturbed during shipping or transport after the samples have been released from the sampler to the shipper (see Figure 4). The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been packaged for shipping (see Figure 5).

Environmental samples: Environmental samples include drinking water, most groundwater and surface water, soil, sediment, treated municipal and industrial wastewater effluent, indoor and ambient air, nonhazardous bulk materials, soil gas, dust, asbestos, and biological specimens. Environmental samples typically contain low concentrations of contaminants and, when handled, require only limited precautionary procedures.

Field Blank: A field blank is any blank sample that is packaged and shipped from the field. Each field blank is assigned its own unique sample number. Field blanks include trip blanks, rinse blanks, and equipment blanks, all intended to assess potential cross-contamination. For example, a trip blank checks for contamination during sample handling, storage, and shipment from the field to the laboratory.

Nonhazardous samples: Nonhazardous samples are those samples that do not meet the definition of a hazardous sample and **do not** need to be packaged and shipped in accordance with the International Air Travel Association's (IATA's) "Dangerous Goods Regulations" (DGR) or U.S. Department of Transportation's (U.S. DOT's) "Hazardous Materials Regulations" (HMR) defined in Title 49 Code of Federal Regulations (CFR).

The following definitions are provided to further distinguish environmental and nonhazardous samples from dangerous good and hazardous samples:

Dangerous goods: Dangerous goods are articles or substances that can pose a significant risk to health, safety, or property when transported by air; they are classified as defined in Section 3 of the DGR (IATA 2014).

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Hazardous samples: Hazardous samples include dangerous goods and hazardous substances. Hazardous samples shipped by air should be packaged and labeled in accordance with procedures specified by the DGR; ground shipments should be packaged and labeled in accordance with the HMR.

Hazardous substance: A hazardous substance is any material, including its mixtures and solutions, that is listed in 49 CFR 172.101 <u>and</u> its quantity, in one package, equals or exceeds the reportable quantity (RQ) listed in Table 1 to Appendix A of 49 CFR 172.101.

1.4 REFERENCES

- General Awareness, H&S contacts, and course training information" click here. (Tetra Tech, Inc., EMI Operating Unit. Intranet) Available on-line at: https://int.tetratech.com/sites/EMI/hs/Pages/Dangerous-Goods-Shipping.aspx
- International Air Transport Association (IATA). 2014. "Dangerous Goods Regulations. 2014." For sale at: http://www.iata.org/publications/Pages/standards-manuals.aspx. Updated annually, with new edition available late in year.
- U.S. Environmental Protection Agency (EPA). 40 CFR, 763 Subpart F, Asbestos Hazards Emergency Response Act (AHERA).
- EPA. 2011. "Contract Laboratory Program Guidance for Field Samplers." EPA 540-R-09-03. Available on-line at: http://www.epa.gov/oerrpage/superfund/programs/clp/download/sampler/CLPSamp-01-2011.pdf. January.

1.5 REQUIREMENTS AND RESOURCES

The procedures for packaging and shipping samples require the following:

- Coolers (insulated ice chest) or other shipping containers appropriate to sample type
- Ice
- Bubble wrap or similar cushioning material
- Chain-of-custody forms and seals
- Airbills
- Resealable plastic bags for sample jars and ice
- Tape (strapping and clear)
- Large plastic garbage bags for lining the cooler
- Temperature blank sample bottle filled with distilled water can be included in the cooler if appropriate to sample type

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• Trip blank samples used to check for volatile contamination during sample handling in the field and shipment from field to laboratory should be included in the cooler if volatile organic compounds are requested for analysis. Also see Field Blank under definitions.

2.0 PROCEDURES

The following procedures apply to packaging and shipping nonhazardous and environmental samples.

2.1 PACKAGING SAMPLES

After they have been appropriately containerized and labeled, environmental samples should be packaged as described in this section. This section covers procedures for packing samples for delivery by commercial carrier (air or ground) and hand delivery of environmental samples (by employee or courier), as well as shipping asbestos and air quality samples. Note that these instructions are general; samplers also should be aware of client-specific requirements concerning the placement of custody seals or other packaging provisions.

2.1.1 Packaging Samples for Delivery by Commercial Carrier (Air or Ground)

Samples shipped by commercial carriers should be packed for shipment using the following procedures and in compliance with all carrier requirements:

Preparing the sample:

- 1. Allow a small amount of headspace in all bottles, or as instructed by the laboratory (except volatile organic compound [VOC] containers with a septum seal) to compensate for any changes in pressure and temperature during transfer.
- 2. Be sure the lids on all bottles are tight (will not leak). Lids maybe taped or sealed with custody seals as added protection or as required.
- 3. Place sample containers in resealable plastic bags.

Preparing the cooler:

- 1. Secure and tape the drain plug of the cooler with fiber or duct tape.
- 2. It is recommended that the cooler be lined with a large plastic garbage bag before samples, ice, and absorbent packing material are placed in the cooler.
- 3. Wrap the sample containers in bubble wrap or line the cooler (bottom and sides) with a cushioning material to prevent breakage of bottles or jars during shipment.
- 4. Add a sufficient quantity of ice to the cooler to cool samples to 4 °C (± 2 °C). Ice should be double bagged in resealable plastic bags to prevent the melted ice from leaking out. If required, include one temperature blank (a sample bottle filled with distilled water) per cooler.

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- 5. For volatile organic analysis (VOA) samples only, include one trip blank for VOA analysis per shipment matrix in each cooler.
- 6. Fill all remaining space between the bottles or jars with bubble wrap.
- 7. Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).
- 8. If more than one cooler is being shipped, mark each cooler as "1 of 2," "2 of 2," and so forth.
- 9. Place the chain-of-custody forms (see Figure 2) into a resealable plastic bag, and tape the bag to the inner side of the cooler lid (see Figure 3). If you are shipping more than one cooler, copy the chain-of-custody form so that there is one copy of all forms in each cooler. The samples listed on the chain-of-custody form must match exactly with the contents of the cooler. Tape any instructions for returning the cooler to the inside of the lid.
- 10. Close the lid of the cooler and tape it shut by wrapping strapping tape around both ends and hinges of the cooler at least once.
- 11. Place two signed custody seals (see Figure 4) on opposite sides of the cooler, ensuring that each one covers the cooler lid and side of the cooler (see Figure 5; note that in contrast to the figure, the seals should be placed on the opposite sides of the cooler and offset from each other, rather than directly across from each other as shown in Figure 5). Place clear plastic tape over the custody seals so that the cooler cannot be opened without breaking the seal.
- 12. Shipping containers must be marked "THIS END UP." Arrow labels, which indicate the proper upward position of the container, may also be affixed to the container (see Figures 3 and 5). A label containing the name, phone number, and address of the shipper should be placed on the outside of the container (Federal Express [FedEx] label) (see Figure 1).
- 13. Ship samples overnight using a commercial carrier such as FedEx.

2.1.2 Hand Delivery of Environmental Samples (by Employee or Courier)

Samples hand-delivered to the laboratory should be packed for shipment using the following procedures:

Preparing the sample:

- 1. Bottles can be filled completely with sample (required for VOC containers with a septum seal).
- 2. Be sure the lids on all bottles are tight (will not leak).

Preparing the cooler:

- 1. Secure and tape the drain plug of the cooler with fiber or duct tape.
- 2. Wrap the sample containers in bubble wrap and/or line the cooler (bottom and sides).
- 3. Add a sufficient quantity of ice to the cooler to cool samples to 4 °C. Ice should be double bagged in resealable plastic bags to prevent the melted ice from leaking out. If required, include one temperature blank (a sample bottle filled with distilled water) per cooler.
- 4. For VOA samples only, include one trip blank for VOA analysis per shipment matrix in each cooler.
- 5. If more than one cooler is being shipped, mark each cooler as "1 of 2," "2 of 2," and so forth.

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- 6. Place chain-of-custody form (see Figure 2) in a resealable plastic bag and tape to the inside of the cooler lid, close the lid, seal with custody seals, and transfer the cooler to the courier (see Figure 3). Alternatively, when samples will be delivered directly to the laboratory, close the cooler and hand-deliver it with the chain-of-custody form. The samples listed on the chain-of-custody form must match exactly with the contents of the cooler.
- 7. Include any instructions for returning the cooler to the inside of the lid.
- 8. Place two signed custody seals (see Figure 4) on opposite sides of the cooler, ensuring that each one covers the cooler lid and side of the cooler (see Figure 5, note that the seals should be placed on the opposite sides of the cooler and offset from each other, rather than directly across from each other as shown in Figure 5). Place clear plastic tape over the custody seals so that the cooler cannot be opened without breaking the seal.
- 9. Shipping containers must be marked "THIS END UP," and arrow labels, which indicate the proper upward position of the container should be affixed to the container (see Figures 3 and 5).

2.1.3 Shipping Asbestos Samples

Asbestos samples shipped by commercial carriers should be packed for shipment using the following procedures and in compliance with all carrier requirements:

- 1.` Place each asbestos sample in a small resealable plastic bag. Place the bags of asbestos samples in a large resealable plastic bag.
- 2. Select a rigid shipping container (FedEx box) and pack the cassettes upright in a noncontaminating, nonfibrous medium such as a bubble pack to prevent excessive movement during shipping.
- 3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.
- 4. Affix custody seals to the top of the cassettes or outer sample bag so that the bags cannot be opened without breaking the seal.
- 5. Insert the chain-of-custody form in the box. Include a shipping bill and a detailed listing of samples shipped, their descriptions and all identifying numbers or marks, sampling data, shipper's name, and contact information.
- 6. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container will be rejected.
- 7. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.
- 8. Hand-carry samples to the laboratory in an upright position if possible; otherwise, choose that mode of transportation least likely to jar the samples in transit.
- 9. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain-of-custody and sample tracking procedures. This information will also help the laboratory schedule timely analysis for the samples when they are received.

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2.1.4 Shipping Air Samples

Packaging and shipping requirements for air samples vary depending on the media used to collect the samples and the analyses required. Sampling media typically include Summa canisters and Tedlar bags for whole air samples, filters for metals and particulate matter, and sorbent tubes for organic contaminants. This section of the SOP provides general guidelines for packaging and shipping air samples collected using these media. The project FSP or QAPP should also be reviewed for any additional project-specific requirements or instructions.

Summa Canister Samples

- 1. Close the canister valve by tightening the knob clockwise or flipping the toggle switch. Replace the brass cap on the canister inlet.
- 2. If a flow controller was used to collect the air sample over a specified time interval, the flow controller should be removed before replacing the brass cap.
- 3. Fill out the sample tag on the canister with the sample number and the date and time of collection. Include the identification number of the flow controller on the sample tag if one was used. Make sure the information on the sample tag matches the chain-of-custody form.
- 4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final Summa canister vacuum readings; Summa canister identification number; and flow controller identification number.
- 5. Package the Summa canister (and flow controller) in its original shipping box with the original packaging material. Tape the box shut and apply custody seals if required. Note: Summa canisters should never be packaged with ice.
- 6. Summa canister shipments typically include several canisters, and may include more than one shipping box. The chain-of-custody form for the shipment should be sealed within one of the shipping boxes.
- 7. Ship the samples by a method that will meet the holding time. Summa canister samples should be analyzed within 30 days of sample collection.

Tedlar Bag Samples

- 1. Close the Tedlar bag by tightening the valve clockwise.
- 2. Fill out the label on the bag with the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.
- 3. Complete the chain-of-custody form.
- 4. Package the Tedlar bag in a shipping box with appropriate packing material. Multiple bags can be packaged in the same box. Tape the box shut and apply custody seals if required. Note: Tedlar bag samples should not be cooled or packaged with ice.
- 5. Tedlar bag shipments may include more than one shipping box. The chain-of-custody form for the shipment should be sealed within one of the shipping boxes.

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6. Ship the samples using priority overnight delivery. Tedlar bag samples should be analyzed within 3 days of sample collection.

Filter Cassette Samples

- 1. Disconnect the filter cassette from the air sampling pump and replace the plastic caps on the inlet and outlet openings.
- 2. Attach a label to the sample that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.
- 3. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.
- 4. Package the filter cassettes in a shipping box (such as a FedEx box). Use an appropriate packing material (such as bubble wrap) to separate the samples and prevent damage.
- 5. Place the chain-of-custody form within the box, seal the box, and apply custody seals if required. Filter cassette samples typically do not need to be cooled, but check the FSP or QAPP for project-specific requirements.
- 6. Ship the samples by a method that will meet the holding time.

Sorbent Tube Samples

- 1. Disconnect the sample tube from the air sampling pump and seal both ends of the tube with plastic caps.
- 2. Complete a sample label that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.
- 3. If the tube is small and the label cannot be attached to the tube, the tube can be placed in a small sealable plastic bag and the label can be attached to the bag or placed inside the bag with the tube.
- 4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.
- 5. Packaging requirements for the sample tubes will depend on the analysis required, and the sampler should check the FSP or QAPP for project-specific requirements (for example, tubes may need to be wrapped in aluminum foil to prevent exposure to light). Packaging containers and methods include (1) shipping boxes (as described under filter cassette samples), (2) small sample coolers filled with double-bagged ice, and (3) small sample coolers filled with blue ice.
- 6. Place the chain-of-custody form within the box or container, seal the box or container, and apply a custody seal if required.
- 7. If coolers are used for shipping, tape instructions for returning the cooler to the inside of the lid.
- 8. Ship the samples by a method that will meet the holding time.

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Polyurethane Foam (PUF) Tube Samples

- 1. Disconnect the PUF tube from the air sampling pump and wrap the tube in aluminum foil.
- 2. Attach a label to the wrapped sample tube that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.
- 3. Wrap the PUF tube in bubble wrap and place the tube in a glass shipping jar.
- 4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.
- 5. Package the PUF tube jars in a cooler that is filled with double-bagged ice. Use bubble wrap or other cushioning material to separate the samples and prevent breakage.
- 6. Place the chain-of-custody form within the cooler, seal the cooler, and apply a custody seal if required.
- 7. If coolers are used for shipping, tape instructions for returning the cooler to the inside of the lid.
- 8. Ship the samples by a method that will meet the holding time. Samples collected in PUF tubes typically must be extracted within 7 days of collection.

2.2 SHIPPING DOCUMENTATION FOR SAMPLES

Airbills, chain-of-custody forms, and custody seals must be completed for each shipment of nonhazardous environmental samples. Figures 1, 2, and 4 provide examples of these forms and instructions for completing them.

Field staff collecting samples should also review their field work plans to confirm what documentation must be completed during each sampling event, including client-specific requirements. For example, some EPA programs have a specific requirement to use Scribe software, an environmental data management system, to create sample documentation, electronically input information into Traffic Report or chain-of-custody forms, and enter other data.

- The Scribe software can be accessed from the EPA Environmental Response Team (ERT) at the following address: http://www.ertsupport.org/scribe home.htm
- The ERT User Manual for Scribe, reference, and training materials can be accessed from the Scribe Support Web site at the following address: http://www.epaosc.org/scribe

Note that some laboratories must routinely return sample shipping coolers within 14 calendar days after the shipment has been received. Therefore, the sampler should also include instructions for returning the cooler with each shipment, when possible. The sampler (not the laboratory) is responsible for paying for return of the cooler and should include shipping airbills bearing the sampler's shipping account number,

as well as a return address to allow for return of the cooler (see Figure 1). Samplers should use the least expensive option possible for returning coolers.

2.3 SHIPMENT DELIVERY AND NOTIFICATION

A member of the field sampling team must contact the laboratory to confirm it accepts deliveries on any given day, especially Saturdays. In addition, samplers should ensure the laboratory has been notified in advance of the pending shipment and notify any additional parties as required. The sampler needs to know the laboratory's contact name, address, and telephone number and be aware of the laboratory's requirements for receiving samples.

The sampler needs to know the shipping company's name, address, and telephone number (see Figure 1). In addition, samplers should be aware of the sample holding times, shipping company's hours of operation, shipping schedule, and pick-up and drop-off requirements to avoid delays in analytical testing.

Priority Overnight Delivery

Priority overnight delivery is typically the best method for shipment. Delays caused by longer shipment times may cause the sample temperature to rise above the acceptable range of 4° C (\pm 2 $^{\circ}$ C) and technical holding may expire, which in turn may compromise sample integrity and require recollection of samples for analysis. If sample delivery procedures are to be modified for particular contract- or laboratory-specific requirements, the procedures should be clearly described in site-specific plans such as work plans, FSPs, or QAPPs.

Saturday Delivery

If planning to ship samples for Saturday delivery, the laboratory must be contacted in advance to confirm it will accept deliveries on Saturdays or arrange for them to be accepted. In addition, samplers should ensure the laboratory has been notified in advance of the pending shipment and notify any additional parties as required.

2.4 HEALTH AND SAFETY CONSIDERATIONS

In addition to the procedures outlined in this SOP, all field staff must be aware of and follow the health and safety practices that result from the Activity Hazard Analyses (AHA) for the project. The AHAs include critical safety procedures, required controls, and minimum personal protective equipment (PPE) necessary to address potential hazards. The hazards specific to project tasks must be identified and

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controlled to the extent practicable and communicated to all project personnel via the approved, project-specific Health and Safety Plan (HASP).

3.0 POTENTIAL PROBLEMS

The following potential problems may occur during sample shipment:

- Leaking package. If a package leaks, the carrier may open the package and return the package. Special care should be taken during sample packaging to minimize potential leaks.
- Improper labeling and marking of package. If mistakes are made in labeling and marking the package, the carrier will most likely notice the mistakes and return the package to the shipper, thus delaying sample shipment. A good practice is to have labels, forms, and container markings double checked by a member of the field team.
- Bulk samples and air samples delivered to the analytical laboratory in the same container. If samples are combined in this way, they will be rejected. Always ship bulk samples in separate containers from air samples.
- Issues in packing asbestos samples. When asbestos samples are shipped, avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials with asbestos samples because of possible contamination.
- Improper, misspelled, or missing information on the shipper's declaration. The carrier will most likely notice these errors as well and return the package to the shipper. A good practice is to have another field tram member double check this information.
- Missed drop off time or wrong location. Missing the drop off time or having the wrong location identified for drop off will delay delivery to the laboratory and may cause technical holding times to expire. Establish the time requirements in advance of completing the field effort and be sure and provide some contingency time for potential delays such as traffic or checking and redoing paperwork.
- Incorrectly packaging samples for analysis at multiple laboratories. For example, inorganic samples may be shipped to one laboratory for analysis, while organic samples may need to be shipped to another laboratory. All field staff should be aware which samples are to be shipped to which laboratory they package samples for multiple types of analysis.
- Holidays or weather-related delays. Be aware of holidays and weather forecasts that could cause
 delays in delivery. Delays caused by longer shipping times may cause technical holding times to
 expire, which in turn may compromise sample integrity or require recollection of samples for
 analysis.
- Not noting field variances in field log book. Field variances should be noted in the field log book and the project manager notified. Common field variances include:
 - Less sample volume collected than planned. Notify appropriate staff and the laboratory to ensure there is an adequate amount for analysis.

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Sample collected into incorrect jar because of broken or missing bottle-ware. Notify
appropriate laboratory staff to ensure there is no confusion regarding the analysis of the
sample.

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FIGURE 1

EXAMPLE OF A FEDEX US AIRBILL FOR LOW LEVEL ENVIRONMENTAL SAMPLES

FedEx. US Airbill	Sender's Copy
From Number Page 10/5/07 Service's Feelix 9999-9999-9 Service's Feelix 9999-9999-9 Service's Feelix 9999-999-9 Service's Feelix 9999-9 Service's Feelix 9999-9 Service's Feelix 9999-9 Service's Feelix 9999-9 Service's Fe	4e Express Package Service RedSx Priod Voernight Strandard Overnight
Address 1234 Main Street Day Phoenix State AZ ZP 85034 2 Your Internal Billing Reference AAA300	FeeEx Toy Freight FeeEx 2Day Freight Substitution FeeEx 2Day Freight FeeEx 2Day Fre
To Recipient's Liam Riley Phone (405) 555-8300 Company Ridgeway Design Recipient's 2020 Vision Street We served deliver to P.D. leases or P.D. &P codes.	SATURDAY Delivery HOLD Wheelingy of Freitz Location
Address To require a package to held at a specific feelfs location, prior feelfs edd-west hore. One Atlanta State GA 219 30305 Ship and track packages at fedex.com Simplify your shipping. Manage your account. Access all the tools you need.	Felia Aux Su. Total Packages Total Weight Total Declared Value 1 1 \$ 450.00 Total Packages Total Signature Total Signature The Act of t

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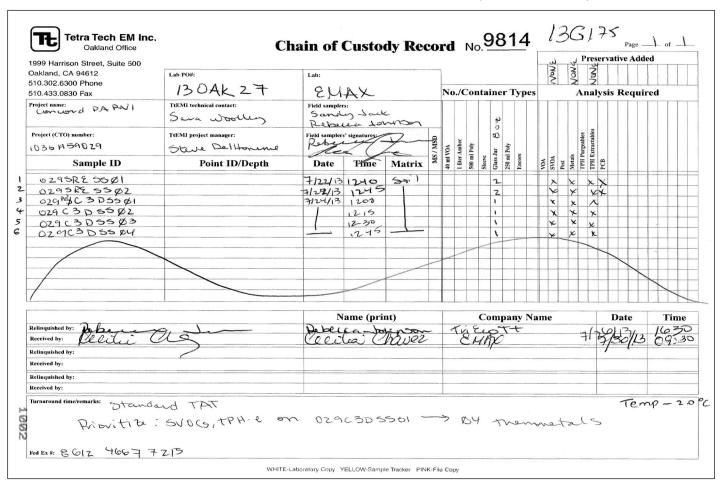
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Filling Out the FedEx US Airbill

- The sender *must complete* the following fields on the pre-printed airbill:
 - Section 1: Date
 - Section 1: Sender's FedEx Account Number
 - Section 1: Sender's Name, Company, Address, and Phone Number
 - Section 2: Internal Billing Reference (Project Number)
 - Section 3: Recipient's Name, Company, Address, and Phone Number
 - Section 4: Express Package or Freight Services (Priority Overnight)
 - Section 5: Packaging (usually "Other," your own packaging)
 - Section 6: Special Handling (Saturday delivery if prearranged with receiving laboratory;
 "No" dangerous goods contained in shipment)
 - Section 7: Payment ("Bill to Sender")
 - Section 7: Total Number of Packages
 - Section 7: Total Weight (completed by FedEx employee)
 - Section 8: Delivery Signature Options ("No Signature Required")

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FIGURE 2
EXAMPLE OF A CHAIN-OF-CUSTODY FORM (WHITE COPY)



Completing a Sample Chain-of-Custody Form

After samples have been collected, they will be maintained under chain-of-custody procedures. These procedures are used to document the transfer of custody of the samples from the field to the designated analytical laboratory. The same chain-of-custody procedures will be used for the transfer of samples from one laboratory to another, if required.

The field sampling personnel will complete a Chain-of-Custody and Request for Analysis (CC/RA) Form (Figure 1, Chain of Custody Record) for each separate container of samples to be shipped or delivered to the laboratory for chemical or physical (geotechnical) analysis. Information contained on the triplicate, carbonless form will include:

- 1. Project identification (ID) (for example, contract and task order number);
- 2. Project Contract Task Order (CTO) number;
- 3. Laboratory Project Order (PO) number;
- 4. Tetra Tech Technical Contact;
- 5. Tetra Tech Project Manager
- 6. Laboratory name;
- 7. Field sampler names;
- 8. Field sampler signature;
- 9. Sample ID;
- 10. Point ID and Depth (Do **NOT** include this information on the laboratory copy of the chain-of-custody (top white copy);
- 11. Date and time of sampling;
- 12. Sample matrix type;
- 13. Sample preservation method; note "NONE" if no preservatives;
- 14. Number and types of sample containers and container capacity;
- 15. Sample hazards (if any);
- 16. Requested analysis;
- 17. Requested sample turnaround time or any special remarks;
- 18. Page of ;
- 19. Method of shipment;
- 20. Carrier/waybill number (if any);
- 21. Signature, name, and company of the person relinquishing the samples and the person receiving the samples when custody is transferred;
- 22. Date and time of sample custody transfer;

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23. Condition of samples when they are received by the laboratory.

The sample collector will cross out any blank space on the CC/RA Form below the last sample number listed on the part of the form where samples are listed.

The sampling personnel whose signature appears on the CC/RA Form is responsible for the custody of a sample from time the sample is collected until the custody of the sample is transferred to a designated laboratory, a courier, or to another Tetra Tech employee for transporting a sample to the designated laboratory. A sample is considered to be in custody when the custodian: (1) has direct possession of it; (2) has plain view of it; or (3) has securely locked it in a restricted access area.

Custody is transferred when both parties to the transfer complete the portion of the CC/RA Form under "Relinquished by" and "Received by" or a sample is left at a FedEx facility pending shipment. Signatures, printed names, company names, and date and time of custody transfer are required. When custody is transferred, the Tetra Tech sampling personnel who relinquished the samples will retain the third sheet (pink copy) of the CC/RA Form. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the CC/RA Form. Receipts of Bills of Lading will be retained as part of the permanent documentation in the Tetra Tech project file.

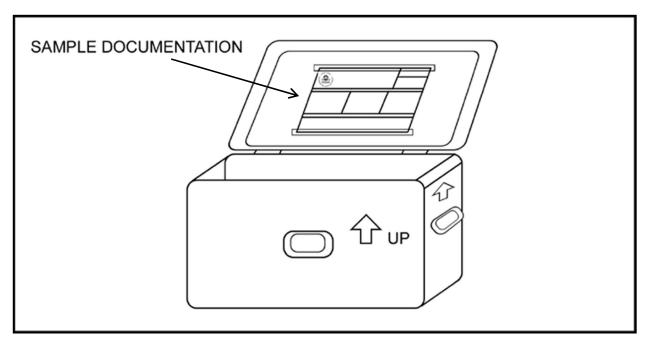
Title: Packaging and Shipping Samples

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FIGURE 3

EXAMPLE OF A SAMPLE COOLER WITH ATTACHED DOCUMENTATION

Place the necessary paperwork (chain-of-custody form, cooler return instructions, and associated paperwork) in the shipping cooler or acceptable container. All paperwork must be placed in a plastic bag or pouch and then secured to the underside of the shipping container lid.



Source: U.S. Environmental Protection Agency. 2011.

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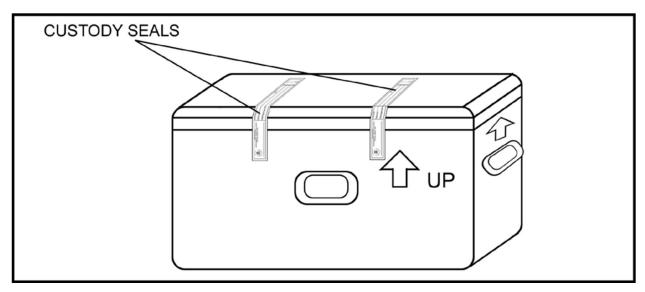
FIGURE 4 EXAMPLE OF A CUSTODY SEAL

Date
Signature

Title: Packaging and Shipping Samples

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FIGURE 5 EXAMPLE OF SHIPPING COOLER WITH CUSTODY SEALS



Source: U.S. Environmental Protection Agency. 2011.

Please note that the two seals typically are affixed *to opposite sides of the cooler and offset from each other*, although the offset is not depicted on the EPA figure above.

SOP APPROVAL FORM

TETRA TECH, INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

RECORDING NOTES IN FIELD LOGBOOKS

SOP NO. 024

REVISION NO. 2

Last Reviewed: November 2014

Carla J Bunks	
	November 24, 2014
Ouality Assurance Approved	Date

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1.0 BACKGROUND

Complete and accurate field documentation is critical to a successful project and the field log book is an important tool to support field documentation needs. The field logbook should include detailed records of all field activities, document interviews with people, and record observations of conditions at a site. Entries should be described in a level of detail to allow personnel to reconstruct, after the fact, activities and events that occurred during their field assignments. Furthermore, entries should be limited to facts. Avoid speculation related to field events and do not record hearsay or unfounded information that may be presented by other parties during field activities. For example, do not record theories regarding the presence or absence of contamination when you are collecting field screening data or speculation regarding the reasons for a property owner's refusal to grant access for sampling.

Field logbooks are considered accountable documents in enforcement proceedings and may be subject to review. Therefore, the entries in the logbook must be accurate and detailed, but should not contain speculative information that could conflict with information presented in subsequent project deliverables and correspondence. Also be aware that the field logbooks for a site may be a primary source of information for depositions and other legal proceedings that may occur months or years after field work is complete and long after our memories have faded. The accuracy, neatness, and completeness of field logbooks are essential for recreating a meaningful account of events.

1.1 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide guidance to ensure that field logbook documentation collected during field activities meets all requirements for its later use. Among other things, field logbooks may be used for:

- Identifying, locating, labeling, and tracking samples
- Recording site activities and the whereabouts of field personnel throughout the day
- Documenting any deviations from the project approach, work plans, quality assurance project plans, health and safety plans, sampling plans, and any changes in project personnel
- Recording arrival and departure times for field personnel each morning and evening and weather conditions each day
- Describing photographs taken during the project.

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In addition, the data recorded in the field logbook may later assist in the interpretation of analytical results. A complete and accurate logbook also aids in maintaining quality control, because it can verify adherence to project scope and requirements.

1.2 SCOPE

This SOP establishes the general requirements and procedures for documenting site activities in the field logbook.

1.3 **DEFINITIONS**

None.

1.4 REFERENCES

Compton, R.R. 1985. Geology in the Field. John Wiley and Sons. New York, NY.

1.5 REQUIREMENTS AND RESOURCES

The following items are required for field notation:

- Field logbooks
- Ballpoint pens or Sharpies with permanent waterproof ink
- 6-inch ruler (optional)

Field logbooks should be bound (sewn) with water-resistant and acid-proof covers, and each page should have preprinted lines, numbered pages, and a single column. They should be approximately $7^{1}/_{2}$ by $4^{1}/_{2}$ inches or $8^{1}/_{2}$ by 11 inches in size. Loose-leaf sheets are not acceptable for use as field notes.* If notes are written on loose paper, they must be transcribed as soon as possible into a bound field logbook by the same person who recorded the notes originally. *Note: Data collection logs and field forms used to record field measurements and data are acceptable as loose-leaf sheets maintained in a three-ring binder with numbered pages.

Ideally, distribution of logbooks should be controlled by a designated person in each office. This person assigns a document control number to each logbook, and records the assignment of each logbook distributed (name of person, date distributed, and project number). The purpose of this procedure is to ensure the integrity of the logbook before its use in the field, and to document each logbook assigned to a

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project. In the event that more than one logbook is assigned to a project, this process will ensure that all logbooks are accounted for at project closeout.

2.0 PROCEDURES

The following subsections provide general guidelines and formatting requirements for field logbooks, and detailed procedures for completing field logbooks.

2.1 GENERAL GUIDELINES

- A separate field logbook must be maintained for each project. If a site consists of multiple subsites (or operable units), designate a separate field logbook for each subsite. Similarly, if multiple activities are occurring simultaneously requiring more than one task leader (well installation, private well sampling, or geophysical survey.), each task leader should maintain a separate field logbook to ensure that each activity is documented in sufficient detail.
- At larger sites, a general field log may be kept at the site trailer or designated field office to track site visitors, document daily safety meetings, and record overall site issues or occurrences.
- Data from multiple subsites may be entered into one logbook that contains only one type of information for special tasks, such as periodic well water-level measurements.
- All logbooks must be bound and contain consecutively numbered pages.
- No pages can be removed from the logbook for any purpose.
- All information must be entered using permanent, waterproof ink. Do not use pens with "wet ink," because the ink may wash out if the paper gets wet. Pencils are not permissible for field notes because information can be erased. The entries should be written dark enough so that the logbook can be easily photocopied.
- Be sure that all entries are legible. Use print rather than cursive and keep the logbook pages free of dirt and moisture to the extent possible.
- Do not enter information in the logbook that is not related to the project. The language used in the logbook should be factual and objective. Avoid speculation that could conflict with information presented in subsequent project deliverables and correspondence (see Section 1.0 above).
- Use military time, unless otherwise specified by the client.
- Include site sketches, as appropriate.
- Begin a new page for each day's notes.
- Include the date at the top of each page.
- At the end of a day, draw a single diagonal line through any unused lines on the page, and sign at the bottom of the page. Note and implement any client specific requirements (for example, some U.S. Environmental Protection Agency (EPA) programs require each logbook page to be signed).

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- Write notes on every line of the logbook. Do not skip any pages or parts of pages unless a day's activity ends in the middle of a page.
- If a line is left blank for some reason, cross out (with a single line) and initial to prevent unauthorized entries.
- Cross out (with a single line) and initial any edits to the logbook entries. Edits should only be made if the initial entry is illegible or erroneous. Do not make corrections for grammar or style.

2.2 LOGBOOK FORMAT

The layout and organization of each field logbook should be consistent and generally follow the format guidelines presented below. Some clients or contracts may have specific formatting guidelines that differ somewhat from this SOP; review client requirements at the start of the project to help ensure any client-specific guidelines are integrated.

2.2.1 Logbook Cover

Write the following information on the front cover of each logbook using a Sharpie or similar type permanent ink marker:

- Logbook document control number (assigned by issuer)
- "Book # of #" (determined by the project manager if there is more than one logbook for the project)
- Contract and task order numbers
- Name of the site and site location (city and state)
- Name of subsite (or operable unit), if applicable
- Type of activity (if logbook is for specific activity, such as well installation or indoor air sampling)
- Beginning and ending dates of activities entered into the logbook

2.2.2 Inside Cover or First Page

Spaces are usually provided on the inside front cover (or the opening page in some logbooks) for the company name, address, contact names, and telephone numbers. If preprinted spaces for this information are not provided in the logbook, write the information on the first available page. Information to be included on the inside front cover or first page includes:

- Tetra Tech project manager and site manager and phone numbers
- Tetra Tech office address

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- Client contact and phone number
- Site safety officer and phone number
- Emergency contact phone number (911, if applicable, or nearest hospital)
- Subcontractor contacts and phone numbers
- Site property owner or property manager contact information

2.3 ENTERING INFORMATION IN THE LOGBOOK

The following lists provide guidance on the type of information to be included in a typical field logbook. This guidance is general and is not intended to be all-inclusive. Certain projects or clients may specify logbook requirements that are beyond the elements presented in this SOP.

General Daily Entries:

- Document what time field personnel depart the Tetra Tech office and arrive at the hotel or site. If permitted by the client to charge travel time for site work, document what time personnel leave and arrive at the hotel each day. (This information may be needed at remote sites where hotel accommodations are not near the site.)
- Indicate when all subcontractors arrive and depart the site.
- Note weather conditions.
- Include the date at the top of each page.
- Document that a site safety meeting was held and include the basic contents of the meeting.
- List the level of protection to be used for health and safety.
- Summarize the day's planned activities.
- Summarize which activities each field team member will be doing.

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Field Activity Entries:

- Refer to field data collection forms for details about field data collection activities (for example time, date, depth of samples, field measurements). If separate field sampling sheets are not used, see section below regarding logbook entries for sampling activities.
- Refer to well purge forms, well construction logs, and other activity-specific forms as applicable rather than including this type of information in the field logbook. These other forms allow the information to be more accessible at a later date.
- List any air monitoring instrumentation used, with readings and locations.
- Refer to instrument field logs for equipment calibration information.
- Summarize pertinent conversations with site visitors (agency representatives, property owners, client contacts, and local citizens).
- Summarize any problems or deviations from the quality assurance project plan (QAPP) or field sampling plan.
- Document the activities and whereabouts of each team member. (As indicated in Section 2.1, multiple logbooks may be required to ensure sufficient detail for contemporaneous activities).
- Indicate when utility clearances are completed, including which companies participated.
- Indicate when verbal access to a property is obtained.
- Include names, addresses, and phone numbers of any pertinent site contacts, property owners, and any other relevant personnel.
- Document when lunch breaks or other work stoppages occur.
- Include approximate scale for all diagrams. If a scale is not available, write "not to scale" on the diagram. Indicate the north direction on all maps and cross-sections, and label features on each diagram.

Sampling Activity Entries: The following information should typically be on a sample collection log and referenced in the log book. If the project does not use sample sheets as a result of project-specific requirements, this information should be included in the logbook.

- Location description
- Names of samplers
- Collection time
- Designation of sample as a grab or composite sample
- Type of sample (water, sediment, soil gas, or other medium)
- On-site measurement data (pH, temperature, and specific conductivity)

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- Field observations (odors, colors, weather)
- Preliminary sample description
- Type of preservative used.
- Instrument readings, if applicable

Closing Daily Entries:

- Describe decontamination procedures (personnel and equipment).
- Describe handling and disposition of any investigation-derived wastes.
- Summarize which planned activities were completed and which ones were not.
- Note the times that personnel depart site for the day.
- Summarize any activities conducted after departing the site (paperwork, sample packaging, etc.).
 This may be required to document billable time incurred after field activities were completed for the day.

Photographic Log Entries:

- For digital photographs, indicate in the text that photographs were taken and the location where the photographs can be found (for example, in the project file).
- Camera and serial #
- Photographer
- Date and time of photograph
- Sequential number of the photograph and the film roll number or disposable camera used (if applicable)
- Direction of photograph
- Description of photograph

2.4 LOGBOOK STORAGE

Custody of logbooks must be maintained at all times. During field activities, field personnel must keep the logbooks in a secure place (locked car, trailer, or field office) when the logbook is not in personal possession. When the field work is over, the logbook should be included in the project file, which should be in a secured file cabinet. The logbook may be referenced in preparing subsequent reports and may also be scanned for inclusion as an appendix to a report. However, it is advisable to obtain direction directly from the client before including the logbook as a report appendix, because its inclusion may not be appropriate in all cases.

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2.5 HEALTH AND SAFETY CONSIDERATIONS

In addition to the procedures outlined in this SOP, all field staff must be aware of and follow the health and safety practices that result from the Activity Hazard Analyses (AHAs) for a project. The AHAs include critical safety procedures, required controls, and minimum personal protective equipment (PPE) necessary to address potential hazards. The hazards specific to project tasks must be identified and controlled to the extent practicable and communicated to all project personnel via the approved, project-specific Health and Safety Plan (HASP).

STANDARD OPERATING PROCEDURE SITE SPECIFIC SOP-29

FIELD PORTABLE X-RAY FLUORESCENCE ANALYSIS OF SOIL SAMPLES

1.0 PURPOSE

This procedure of this SOP is for the semiquantitative analysis of metallic lead particles and chemical compounds of lead in soil using a field portable x-ray fluorescence (FPXRF) spectrometer. This procedure is based on the United States Environmental Protection Agency (EPA)-approved XRF field screening method for elemental analysis (Method 6200). Use of the Innov-X alpha series XRF is planned for this site investigation.

2.0 SCOPE, APPLICATION, AND LIMITATIONS

2.1 Scope of Procedure

Although it is possible to use FPXRF to measure analytes in situ, this Standard Operating Procedure (SOP) requires removal of a soil sample from its native environment prior to analysis. By removing, drying, and homogenizing the sample prior to analysis, more precise and accurate results are obtained.

2.2 Analyst Training

Use of this method is restricted to personnel both trained and knowledgeable in the operation of the Innov-X alpha series XRF instrument or under the supervision of a trained and knowledgeable individual. Proper training for the safe operation of the instrument should be completed by the analyst prior to analysis. This training may be obtained directly from INNOV-X, an INNOV-X instrument distributor or lessor, or trained Tetra Tech personnel.

3.0 ACRONYMS AND ABBREVIATIONS

FPXRF: Field portable x-ray fluorescence.

mg/kg: milligrams per kilogram.
MDL: Method detection limit.

PQL: Practical quantitation limit.

QC: Quality control.

RPD: Relative percent difference.

USGS: United States Geological Survey.

XRF: X-ray fluorescence.

4.0 RESPONSIBILITIES

Analyst/Chemist - Responsible for all aspects of sample preparation and analysis, including equipment maintenance. Responsibility also includes maintaining chain-of-custody of samples after receipt from sampling personnel.

5.0 PROCEDURES

5.1 Safety

5.1.1 Radiation Safety

Radiation safety practices for the INNOV-X instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or anyone other than the manufacturer.

An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.1.2 Protective Equipment

Analysts must wear disposable plastic gloves when sample aliquots are being transferred from one vessel to another. Consult the health and safety plan for other protection requirements.

5.2 **Apparatus and Materials**

Apparatus and materials consist of the following:

INNOV-X Alpha Series FPXRF spectrometer with data processing unit (iPAQ) pocket personnel computer.

INNOV-X Alpha Series XRF instrument manual to match the INNOV-X Alpha Series instrument.

<u>Aluminum drying pans or aluminum foil</u>: Sized suitably to hold as much as 50 grams of sample and fit into the drying oven.

Calibration verification check sample: A National Institute of Standards and Technology (NIST) or other standard reference material (SRM) that contains lead in a concentration range that is compatible with the project objectives to verify the accuracy of the instrument. SRMs can be obtained from NIST, the U.S. Geological Survey (USGS), or the Canadian National Research Council. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. Acceptable limits for SRM percent recoveries are usually provided with the SRM. In their absence, a limit of ± 30 percent will be used as a guideline.

<u>Instrument Blank</u>: May be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate.

Lead calibration check standard: Supplied by the FPXRF manufacturer.

Method blank material for performing method blank checks: May be lead-free silica sand or lithium carbonate that undergoes the same preparation procedure as the samples.

Battery charger.

<u>Polyethylene sample cups</u>: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

X-ray window film: Mylar[™], Kapton[™], Spectrolene[™], polypropylene, or equivalent; 2.5 to 6.0 micrometers (μm) thick.

Sample containers: glass or plastic to store samples.

<u>Sieves</u>: 60-mesh Stainless steel, Nylon, or equivalent for preparing soil and sediment samples if necessary.

<u>Trowels</u>: for collecting soil samples.

<u>Plastic bags</u>: used for collection and homogenization of soil samples. May also be used as sample presentation device.

<u>Drying oven</u>: standard convection or toaster oven, for soil samples that require drying.

Rolling pin (optional): Wooden rolling pin for crushing samples.

5.3 Sample Collection, Preservation, and Handling

Samples should be provided to the FPXRF analyst in plastic bags. The analyst is responsible for maintaining chain-of-custody of all samples until all analyses have been successfully completed. No sample preservation is necessary. All samples should be handled in accordance with sample handling SOPs in effect for the field event.

5.4 Preventive Maintenance

Refer to the instrument manual for specific manufacturer's recommendations.

5.5 <u>Instrument Start-Up</u>

- 5.5.1 Ensure the pocket PC (iPAQ) is plugged into the FPXRF instrument body and install a fully charged battery into the instrument.
- 5.5.2 Press the ON/OFF button on the base of the pistol grip of the instrument. If the iPAQ does not automatically power up, press the Power button in the right corner of the iPAQ.
- 5.5.3 Tap the Microsoft icon at the upper left corner of the iPAQ.
- 5.5.4 Chose START.
- 5.5.5 Tap "Soil Mode" on the menu or choose Mode (bottom of screen) and then choose Soil Mode from the drop down menu.
- 5.5.6 Allow the instrument to warm up (approximately 3 minutes).
- 5.5.7 Release the manual trigger lock.
- 5.5.8 Standardize the instrument in accordance with Section 5.6.

5.6 Standardization/Calibration Check

Analyses of samples should not be started if the instrument has not been standardized. To verify proper calibration of the instrument it is necessary to periodically complete the automated standardization procedure. This must be done any time the instrument is restarted and every 4 hours of operation, although re-standardization may be done at any other time (e.g., when instrument drift is suspected).

- 5.6.1 Click the standardization piece (supplied with the instrument) on the front of the instrument, verifying that the solid portion of the standard completely covers the analysis window.
- 5.6.2 Select "Tap here to Standardize" or select File → Standardize. The red light on top of the instrument will blink indicating that the instrument is producing x-rays and the shutter is open. The amber light on the rear of the instrument will also be illuminated and a status bar will appear to display the progress of the standardization.
- 5.6.3 Upon successful standardization the message "Successful Standardization" will appear along with the instrument resolution. Tap "ok" to dismiss the completion message. If problems are encountered, either follow the prompts that appear or repeat the standardization. Note any error messages that appear as they may be useful if the instrument manufacturer must be contacted. Additional assistance is also available in the manufacturer's instrument manual.

5.7 Quality Control

The quality control (QC) program includes analysis of blanks, calibration verification checks, duplicate analyses, and field duplicate samples. For all the above areas, any identified problems and corrective action must be documented in the instrument run log, analysis narrative report, and instrument maintenance log or standards log (as applicable). Identical operating conditions will be used for each sample.

5.7.1 Laboratory Blanks

Two types of blank samples should be analyzed for FPXRF analysis: instrument blanks and method blanks.

5.7.1.1 At the beginning of each day, at the end of each day, and after every 20th sample or when potential contamination of the instrument is suspected, analyze an instrument blank to verify that no contamination exists in the spectrometer or on the probe window.

If the lead concentration in the blank exceeds the method detection limit (MDL) (see Section 5.9.3), check the probe window and other potentially contaminated instrument components for contamination. If contamination is not causing the elevated blank readings, re-standardize the instrument according to manufacturer's instructions.

5.7.1.2 After every 20th sample analyze a method blank. If the method blank lead concentration exceeds the practical quantitation limit (PQL, see Section 5.9.4), identify the cause of the elevated lead concentration and reanalyze all samples since the last acceptable method blank.

5.7.2 Calibration Verification Checks

- 5.7.2.1 After performing each blank check (Section 5.7.2), analyze a calibration verification check sample to check the accuracy of the instrument and to assess the stability and consistency of the analysis.
- 5.7.2.2 If the measured lead percent recovery (See Section 5.9.1) is less than 60 percent or greater than 135 percent, reanalyze the check sample. If the value continues to fall outside this acceptance range, the instrument should be re-standardized according to the manufacture's instructions and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

5.7.3 XRF Duplicate Samples

XRF duplicate samples are two portions of the same sample that have been prepared and homogenized together, and then split and analyzed in the same manner by the XRF analyst.

- 5.7.3.1 Analyze an XRF duplicate at a frequency of 1 per 20 or once per day, whichever is more frequent.
- 5.7.3.2 If the computed RPD (See Section 5.9.2) exceeds 50 percent reanalyze both samples. If the RPD again exceeds 50 percent RPD consider whether the high degree of imprecision is caused by sample heterogeneity or other causes. This assessment may be aided by repeating the analysis of a sample that was analyzed previously. If the observed imprecision is attributed to sample heterogeneity, increase the number of readings made per sample to try to limit the imprecision and repeat the analyses.

5.8 <u>Sample Analysis</u>

Note:

This section provides sample analysis instructions, assuming that appropriate instrument start-up and calibration checks have been completed. The longer the instrument count time, the lower the detection limits and the less uncertainty there is with a recorded result. Count time is user-selectable through the instrument's software. When the XRF data will be used in a screening capacity to make preliminary decisions concerning the soil concentrations relative to 400 mg/kg, it is not necessary to obtain a high degree of accuracy or precision with the instrument. In that situation, count times should be limited to less than 60 seconds unless a usually high degree of precision is expected. To change the count time, select Options → Setup Testing and enter the same value (in seconds) to minimum and maximum count times.

Note:

Section 5.7 identifies the appropriate frequencies for conducting various QC sample analyses and the associated acceptance limits and corrective actions for potentially unsuitable conditions. The specified QC analysis frequencies are minimum frequencies. More frequent QC sample analyses are permitted, especially when diagnosing quality problems.

- 5.8.1 Ensure that calibration checks and blanks have been analyzed according to Sections 5.6, 5.7.1, and 5.7.2. Count times should be at least 30 seconds but generally less than 60 seconds.
- 5.8.2 Acquire enough soil sample to fill an 8-ounce jar and separate from it all particles greater than the size of a pea.
- 5.8.3 Homogenize the remaining finer grained portion of the sample by simple mixing until it appears as uniform in texture and composition as practicable. Mixing may be done in a beaker or other suitable lead-free container. If the sample is moist and has high clay content, it may be kneaded in a plastic bag. Mixing should continue for at least 2 minutes to ensure that the sample is well mixed. To aid mixing, the sample may be placed into a thick-walled (3 mil or thicker) gallon-sized freezer bag (e.g., ZipLoc) and rolled flat with a rolling pin to break up large chunks of dirt.
- 5.8.4 Place approximately 20 to 50 grams (one U.S. nickel weighs about 5 grams) in a suitable container (e.g., aluminum drying pan) for drying.

- 5.8.5 Dry the homogenized sample from Step 5.8.4 for approximately 20 to 30 minutes in the oven at a temperature not greater than 150°C (a setting of approximately 300°F). If the sample is not visibly dry after this initial drying time, place the sample back into the oven until the sample is dry.
- 5.8.6 Re-homogenize the dried sample aliquot in a beaker or other suitable lead-free container to obtain a well mixed soil sample. Mixing should continue for at least minute.
- 5.8.7 Place a portion (approximately 1.5 cubic inches) of the dried, homogenized sample aliquot into the instrument manufacturer's recommended sample cup (e.g., a 31.0-mm polyethylene sample cup (or equivalent) or place it in a thin-walled (1.0 mil or thinner) plastic sandwich bag.
- 5.8.8 If using a disposable plastic sample cup, ensure the cup is at least three-quarters full and cover with mylar (or other) film per the manufacturer's recommendations.
- 5.8.9 Present the sample to the instrument in Soil Mode.
- 5.8.10 Perform a single pull of the trigger to start the count. The count time should be the same as was used for the calibrations, calibration checks, and blank analyses. The message "Test in progress" will appear on the instrument and the red light on top of the instrument and will illuminate.
- 5.8.11 When the predetermined count period has expired the message "Test complete" will appear on the instrument. A slight delay may also occur during which time the message "calculating" may appear to indicate that results are being computed.
- 5.8.12 Record the displayed results for lead concentration in mg/kg and the associated error on Figure 1 (XRF Field Form).

CAUTION

Inconsistent positioning of samples in front of the probe window is a potential source of error because the X-ray signal decreases by the square of the distance from the radioactive source. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample surface should be flat and smooth to provide a good contact surface.

- 5.8.13 Remix the sample in the plastic bag (or rotate the sample cup approximately one-third of a turn) then acquire another measurement by repeating Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1.
- 5.8.14 Repeat Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1.
- 5.8.15 Based on the degree of precision demonstrated by the three individual measurements, determine whether additional readings should be acquired on the sample. This determination should be based on professional judgment of the FPXRF analyst and should consider the degree of precision observed during calibration checks and previous sample analyses. The objective is to ensure that the average reading reported for each sample is representative of the true sample concentration. If the analyst feels that non-representative readings are being obtained the analyst should correct the analytical system prior to continuing with analyses.
- 5.8.16 Ensure that measured results are reported to the following standards
 - Results < 1,000 mg/kg (or parts per million) are reported to two significant figures and results
 > 1,000 mg/Kg are reported to three significant figures.
 - All values < MDL should be reported as the MDL and flagged with the letter "U".
 - All values > MDL and < PQL should be reported as is and flagged with the letter "J".

5.9 Calculations

5.9.1 <u>Percent Recovery</u>: The equation for determining percent recovery of calibration verification check standards and standard reference materials is:

$$%R = \frac{Experimental Concentration}{Certified or Known Concentration} X 100 %$$

5.9.2 <u>Relative Percent Difference</u>: The equation for determining relative percent difference for laboratory and field duplicate samples is:

$$RPD = \frac{\left| Amount in Sample 1 - Amount in Sample 2 \right|}{0.5 (Amount in Sample 1 + Amount in Sample 2)} X 100 \%$$

5.9.3 <u>Method Detection Limit (MDL):</u> Because the analyses governed by this SOP are semiquantitative, the manufacturer-specified detection limit will be reported as the MDL unless the specified detection limit is less than 20 mg/kg. Care will taken to ensure that the appropriate count time is consistent with the reported detection limit. However, no value less than 20 mg/kg will be reported as an MDL.

5.9.4 Practical Quantitation Limit (PQL): Multiply the MDL by 3 to obtain the PQL: PQL = MDL*3

6.0 REFERENCES

Innov-X Systems, Inc. Innov-X Systems X-Ray Fluorescence Spectrometers Instruction Manual. Woburn, MA. June 2002.

Stephen Shefsky, NITON Corporation. Comparing Field Portable X-Ray Fluorescence (XRF) To Laboratory Analysis of Heavy Metals In Soil. Presented at the International Symposium of Field Screening Methods for Hazardous Wastes and Toxic Chemicals. Las Vegas, Nevada. January 29-31, 1997.

EPA (U.S. Environmental Protection Agency), Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Office of Solid Waste, Washington, D.C. February 2007

EPA, Region I, Northeast Waste Management Officials' Association (NEWMOA) Technology Review Committee Advisory Opinion. Innovative Technology: X-Ray Fluorescence Field Analysis. September 21, 1999.

FIGURE 1

FIELD PORTABLE XRF SAMPLE ANALYSIS LOG SHEET

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		ERROR AVERAGE															
Project Site Name: Project No.: Analyzed By: SAMPLING AND FIELD PORTABLE XRF RESULTS DATA:		ERROR RUN 3															
		ERROR RUN 2															
		ERROR RUN 1															
		RESULTS AVERAGE															
		RESULT RUN 3															
	LTS DATA:	RESULT RUN 2															
			RESULT RUN 1														
		ELEMENT ANALYSIS															
	LE XRF RESUI	ANALYSIS TIME															
	FIELD PORTAB	ANALYSIS DATE															
Project Site Name: Project No.: Analyzed By:	SAMPLING AND F	SAMPLE ID															